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OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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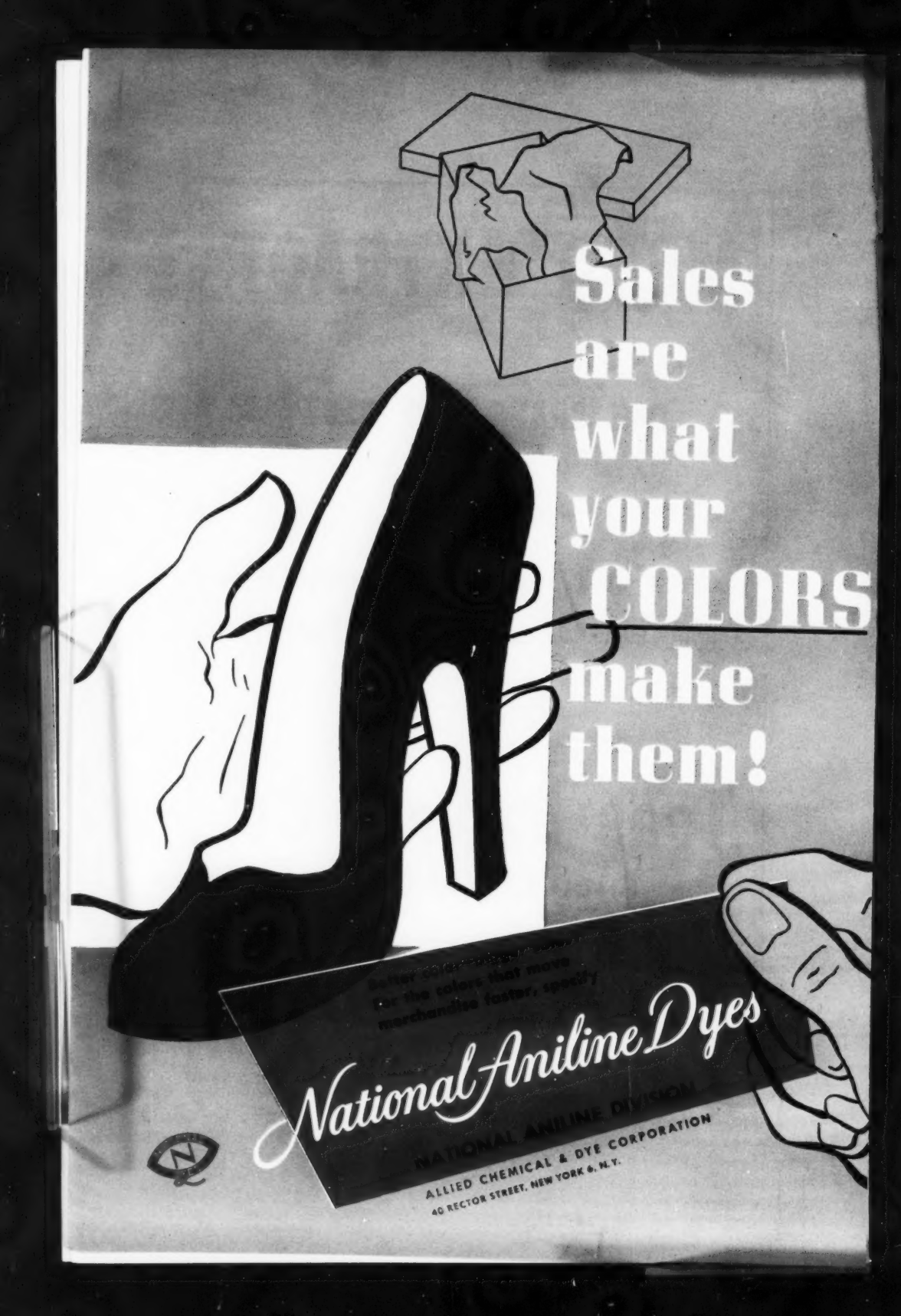
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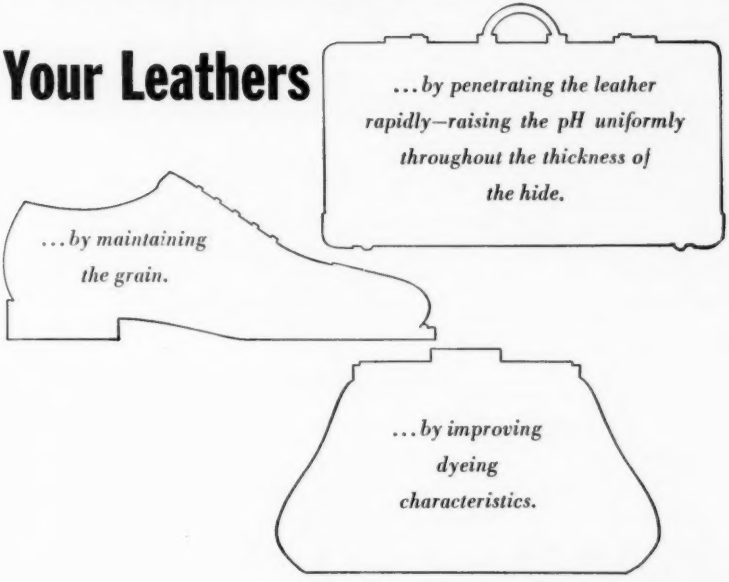
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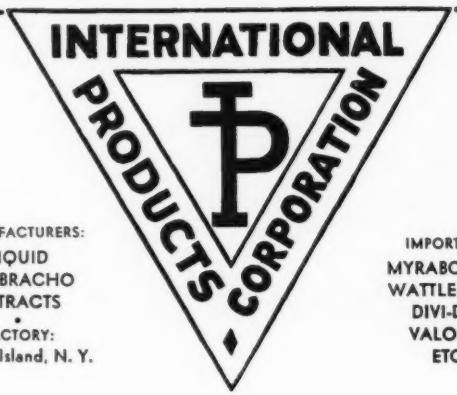

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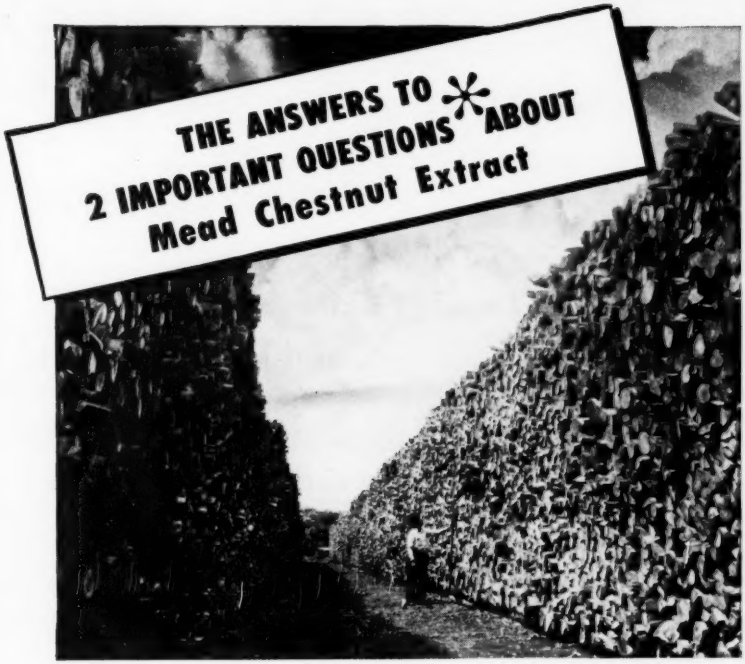

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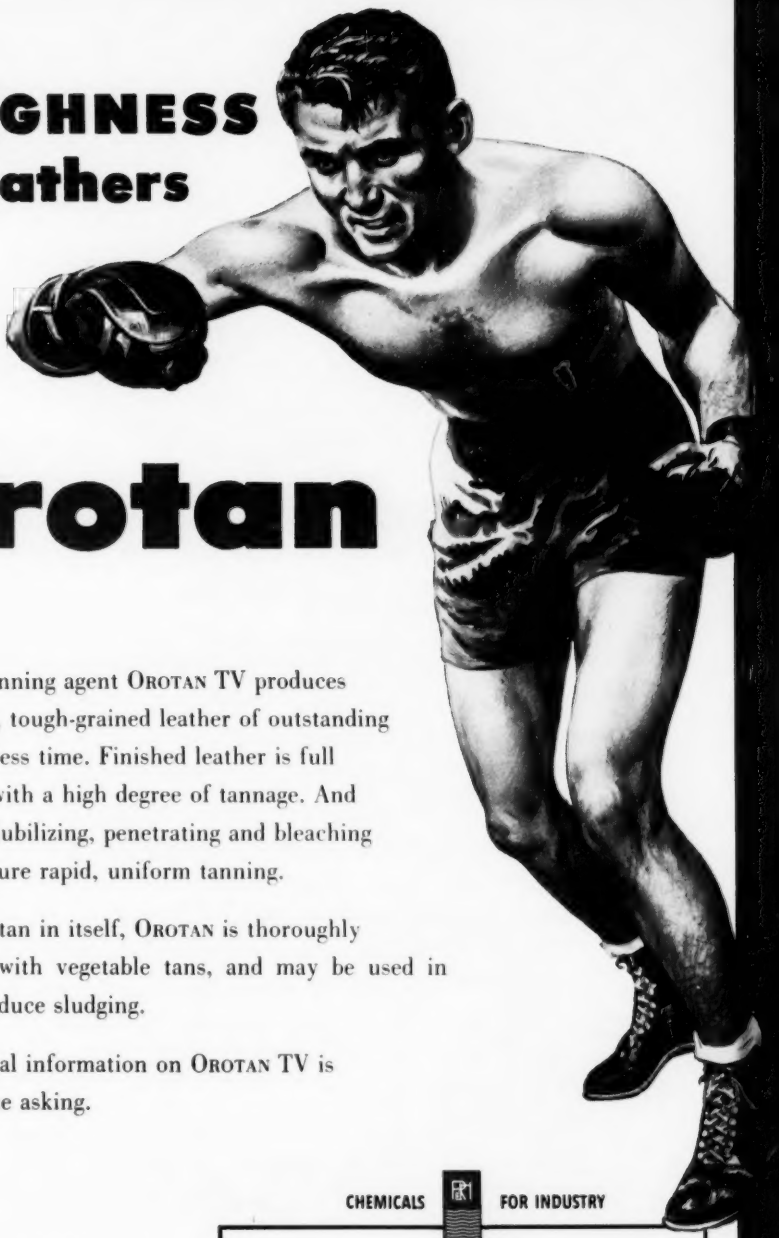

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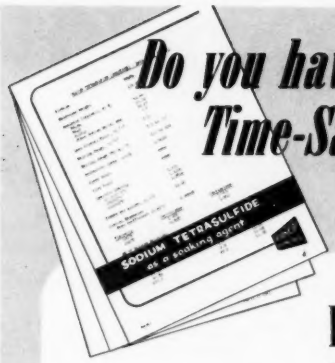

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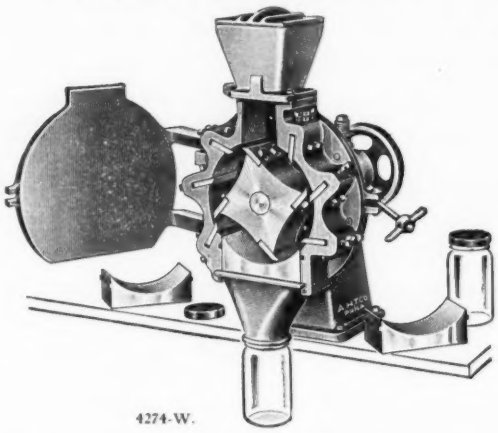

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REPORT OF AUDITING COMMITTEE

We have reviewed the books relative to the financial condition of the American Leather Chemists Association as of June 6, 1950 and have found them to be consistent with the report made by Mr. Arthur W. Holmes, Public Accountant, in his audit of December 31, 1949.

The only differences noted were those required by the normal operating procedures of the Association.

Respectfully submitted,

(Signed)
E. D. Compton
A. N. Kay

Stream Pollution and the Leather Industry*

by E. B. Thorstensen

S. B. Foot Tanning Company
Red Wing, Minnesota

What do we mean by pollution? The laws of Minnesota define pollution as follows: "Pollution means the contamination of any waters of the state so as to create a nuisance or render such waters unclean, or noxious, or impure so as to be actually or potentially harmful or detrimental or injurious to public health, safety or welfare, to domestic, commercial, industrial or recreational use, or to livestock, wild animals, bird, fish, or other aquatic life". Other states are essentially the same.

Under this definition we of the tanning industry are guilty; indeed we have suffered our plants to become synonymous with stench, filth, putridity and with a callous disregard for the rights of others. This reputation has been intensified by several centuries of poor public relations. Of course, it is possible to point to a great many other industries and claim they are worse sinners than we are. For example, in a list of 24 industries, the Chicago Sanitary District places tanneries as number 9, but the more explicit wording of the text states, "the wastes from the Packing Houses and the Stockyards are the most important; next in order are the breweries, followed by the yeast and vinegar houses, the tanneries, and the by-products of coke". This would seem to place tanneries fourth. However, it matters little whether the tanneries take fourth or ninth prize as nuisances: the fact is that we are high on that list.

*Presented at the Forty-Sixth Annual Meeting of the American Leather Chemists Association, French Lick, Indiana, May 31, 1950.

The idea of clean streams is by no means new. Without going back too far we find that the first resolution introduced into the Minnesota Legislature on the subject was presented by Mr. Otto Kohlshorn of Red Wing in 1923. After a few years the sportsmen's clubs, particularly the Izaak Walton League, got back of the idea. The only thought then was of better fishing, hunting and recreation. Over and beyond this first idea, we, as tanners, have many other angles to consider, primarily the fact that as an industry our most necessary material is water,—pure, clean water and lots of it.

If we want to go back further, we find that as early as 1895 the Lawrence Experiment Station of the Massachusetts State Board of Health worked on tannery waste disposal. This work was carried on for fourteen years after which individual tanneries carried on work without finding a satisfactory solution.

The U. S. Public Health Service says the average Eastern tannery uses 8 gallons of water per pound hide. The State of Pennsylvania says 6.5. The average of midwestern tanneries uses 11.5, running as high as 14.

I do not intend to go into statistics, but these figures provide you with a starting point when you get down to the serious side of the question. The days of idealistic thoughts of fishing and hunting are gone and realism in the form of urgent necessity is upon us. Of the 48 states 45 now have pollution abatement legislation, and the Water Pollution Control Act of the 80th Congress is intended to assist the states in pollution abatement.

Water is our most valuable resource. It is not inexhaustible, as New Yorkers have recently come to know, as those in the dust bowl of our wheat country know, as those who draw water from wells are finding with each succeeding year. The conservation of water through pollution abatement is an economic necessity. It is indeed necessary in the interest of public health. You and I are also a part of that public.

The State of Pennsylvania has done an excellent job on the whole stream pollution problem though it probably has a greater job to do than most states, but Pennsylvania, like all other states, has been set back a decade by World War II. The Tannery Waste Disposal Committee of Pennsylvania's Sanitary Water Board reported a study, 1924–1930, which was published in 1931 [3]. This is a helpful and enlightening report. A paper by Oberlander [4] in 1932, and a paper published by the Association's committee on Stream Pollution by Mr. Reuning [7] in 1943 are all worthy of your consideration. While Mr. Reuning's paper covers a survey of 31 tanneries and gives an abundance of information, most of it covers vegetable tanning.

It is a long time between 1923 and 1931. It's a longer time from 1931 to 1943, but with the war over and the immediate emergency set aside, we blossom out in full with the 1947 meeting of the American Chemical Society at Atlantic City.

Papers were presented by Thos. Parran [5] of the U. S. Public Health Service, Abe Wolman [9] of John Hopkins University, Sheppard T. Powell [6], Baltimore,

Maryland, Harlow and Powers [1], Dow Chemical Co., Midland, Michigan, and Robert Sutherland [8], A. C. Lawrence Leather Co., Peabody, Massachusetts. The complete papers by these and many others, covering all sorts of industrial wastes were published in full in the May 1947 issue of Industrial and Engineering Chemistry.

I am informed that all tanneries in Pennsylvania known to the Bureau of Sanitary Engineers have treatment works except one, and that is now under orders from the Sanitary Water Board.

The 1949 Michigan legislature has made more effective its stream pollution laws. The Oregon State Sanitary Authority has issued a flat ultimatum to paper and pulp mills in the Willemette valley to stop polluting practices by the end of 1951.

During 1947–48–49 a very energetic publicity campaign was carried on in Illinois which culminated in the passage of a number of amendments to the Sanitary water laws by the legislature.

As we go into 1950 we are brought sharply face to face with reality. Focusing our attention is the proposed ordinance of the Chicago Sanitary District. Passage of this ordinance, as written, will throw a heavy burden on Chicago tanners. Hearings on this ordinance have brought forth briefs for the tanners by Irving Glass and Dr. Fred O'Flaherty. While we hope these briefs will help change the wording somewhat, the fact still remains that it is going to cost money, a great deal of worry and inconvenience.

To show what can happen, listen to these excerpts from the proposed ordinance—"it shall be unlawful . . . . to discharge into sewers in a twelve month period an aggregate exceeding 3,650,000 gallons".

If our premise of 8 gallons per pound of hide is anywhere near right, and if the tanner is soaking one car of hides per day, this maximum will be exceeded in nine days.

The proposed ordinance reads: "it shall be unlawful to discharge into sewers the following: grease, hair, hides, lime sludge, mineral acids, etc." How many tanneries can escape these?

Permits will be issued for greater quantities at the following prices $8.03 per million gallons in excess of basic 3,650,000 plus $8.07 per ton suspended solids in the excess plus $2.80 for each 1,000 pounds B. O. D. of excess waste mixture.

Example of annual cost based on one car hides per day		
260 days at 4,000,000 gallons is		
104 million excess gallons	104 x 8.03	$835.12
104 million gallons 884 mill. lbs. water		
1200 parts per million	530 tons at 8.07	$4277.10
B.O.D. is only 600 p.p.m.	265 tons at 2.80	$742.00
		$5854.22

The money cost of this would probably not scare very many, but strict enforcement of such a law would put quite a few tanners out of business, together with other industries, as the issuance of a permit does not authorize the discharge of any of the aforementioned substances into the sewers.

At this point let us bring the A.L.C.A. into the picture again. In 1941 D. F. Maskey [3] and R. W. Sarber reported a study made at the Tanners' Council Laboratory on Heavy Beam House effluents. Your committee on stream pollution published a second report in October 1944 containing about 120 abstracts mostly dealing with tannery effluents and their treatment. Mr. Reuning reminds me that in 1920 the Council issued a booklet "What tanners should know about sewage disposal". In January of this year a report was issued, together with recommendations to Council. These recommendations merit a great deal more attention than mere publication in the Journal.

Part of this convention program is inspired by this report, and I sincerely hope that between the A.L.C.A. Council and the Tanners' Council a constructive program of publicity and helpful aids may at last be instituted. In most cases the trouble is that our Journal does not usually reach top management for serious perusal. It is evident that if we are to be of help to our industry, we will have to do a better job

We will have to familiarize ourselves with State and local requirements, bearing in mind that present laws will be stiffened from time to time as public demand for the elimination of pollution mounts. Acquaint yourself with control agencies. They are willing and able to help you and will cooperate if they believe you are sincere. You cannot beat public opinion, and public opinion is demanding clean streams for many reasons.

The desecration of our lakes and streams is an indictment of us all. Our children and children's children certainly merit a heritage better than we are leaving them. When the poet's words about the beauty of nature become a mockery, when our very life and existence are dependent upon clean waters, no amount of protest by commercial interests will stop demands for abatement of these deplorable conditions.

The conditions under which community or industrial wastes may be discharged to a water course are determined by the use which is to be made of the stream or body of receiving water; specifically, whether it is to be used for bathing, recreation or as a raw water supply and or for the further purpose of maintaining water pure enough to support aquatic life, fish life, etc., as well as from an aesthetic standpoint.

In most cases, particularly in communities having no health or sanitation facilities, the local, county or state Health Department is charged with the duty of administering such laws as are provided regarding stream sanitation. This duty is mandatory upon them, not permissive.

The problems of all tanneries are not identical. For one thing, the vegetable or heavy leather tanners have one type of effluent and more is probably known

about their problem than the entirely different situation prevailing in chrome or light leather tanneries.

The location of the plant will also make for different requirements. Perhaps the following will indicate what I mean: some are in small towns; some are in metropolitan areas; some are on tidewater; some are located on the Great Lakes; some on interstate rivers such as the Ohio, Mississippi and others; and some on smaller streams.

In smaller towns a disposal plant capable of handling the normal human needs may not be able to handle the effluent of your plant, and in spite of the fact that you may be the source of a considerable part of the income of the community, you will not be popular unless you cooperate to eliminate the nuisance.

This is the practice in a number of Minnesota cities such as Austin, where the Hormel Packing Company pays a fixed rental as well as a maintenance charge. At South St. Paul two packing houses pay 65 per cent of construction and 90 per cent of operation and maintenance.

Various methods of payment based on different requirements are found in different communities. These are too numerous to mention in this talk so you will have to find out about your own situation if you are not familiar with it.

At Rochester, Minnesota, the municipal plant could not handle the effluent from a food processing firm. The company had to run a pipe line several miles along a railroad right of way to a plant of their own outside the city limits.

In cities like Milwaukee, where an intercepting sewerage system is in operation and where the municipal plant is an activated sludge affair producing fertilizer, the problem is fairly simple. It requires only the screening of the coarser suspended or floating solids and pumping of the effluent into the system.

In some of the small communities in Wisconsin, industry has helped pay for the building of treatment works and for maintaining them through payment for treatment at a reasonable predetermined rate on an equitable basis. The arrangement has merit and through such co-operative effort much can be accomplished.

Obviously, if you have to provide your own disposal system, you are fortunate if you have cheap land available that is adjacent to your plant, but regardless of what your situation is you should first know as much about your own effluent as possible. Next, enlist the services of a good sanitary engineer. He will not only know the local requirements but he can advise you as to the best and most economical method of approach; he can prepare plans and estimates for you, and if you wish, will even supervise construction and start operation.

I believe that much can be accomplished through cooperation between industry and the general public. The officials who administer the laws set up at the instigation of the public have a tough job on their hands because the law is often difficult to enforce. However, we must have industry and the solution to the problem of stream sanitation lies in establishing reasonable, equitable laws relating to all pollution control. Communities and industry alike must recognize waste treatment as a functional or operating charge which is to be assumed by that community or industry. This cannot be accomplished overnight, nor in the accomplishment are we likely to make mountain streams of creeks, rivers or lakes in highly industrialized areas.

One of the first things an industry looking for a location investigates is the water supply. They do not want the polluted effluent of an upstream plant or community, nor do the people in downstream communities cherish using water unfit for use. It is said that the waters of the Ruhr in Germany are used 22 times before emptying into the sea. This would not be possible were it not for adequate treatment plants. The same is true in England where highly industrialized sections, thickly populated, are dependent on water being used over and over again. In two trips through these areas I was impressed with the frequency of these treatment installations along our route. In this respect we are shamefully behind Europe. Our prodigal waste of natural resources has endangered our very existence. Besides abating steam pollution we must see that our watersheds are replanted and our swamps and sloughs, so inadvisedly drained, are restored. Each year we have to go deeper and deeper for water. Communities and industries forced by polluted streams to drill more and more wells are rapidly lowering the water table. Our country is drying up.

For example, in our own particular tannery twenty years ago, one 500 foot flowing well with a 30 foot tail pipe furnished all the water we needed. We soon had to install a deep well pump taking water from 100 feet down. Later another well was necessary, going down to the Hinkley standstone. This well is equipped with a pump drawing from 150 feet down and occasionally loses suction due to abnormal draw down of a steadily falling water table. The city of Red Wing is drilling a new well to 600 feet, which will probably draw from the same strata.

Due to the pollution of the Mississippi River above the Twin Cities Minneapolis hotels and industries are drilling more and more wells and in that area are going down to 1800 feet.

The Twin Cities disposal plant has removed a great deal of pollution from the Mississippi but until all communities and industries along this immense waterway follow suit, it will still remain a great open sewer.

What is the economic implication of all this as far as tanners are concerned? It will be necesssary to recognize that sewage disposal and maintaining water supply are a part of the cost of making leather.

The investment in a treatment plant is capital required for seemingly non-productive purposes, but we can no longer point to others saying they are worse than we are and the day of fixing it with local politicians is fast passing. If I may venture a guess, I would say, that barring a national emergency, in five years time the idea of heavy fines and cease and desist orders will be common in cases of failure to comply.

If you are betting on lower taxes in the future, you might fight for amortization of this investment over a shorter period of time, but if you believe with many industrialists that taxes will continue high, it will be well to have this spread over a longer period. It might also be well to consider that amortization on replacement costs will rise and an investment today of $100,000 will soon take $125,000 to replace.

We must realize the highly competitive situation in which the tanning industry finds itself today not only within itself but with substitutes. We will have to pay particular attention to our individual situation so as to avoid unnecessary or ill-advised expenditures for equipment that will still leave us out on the limb as far as the public is concerned. Too little can be just as wasteful as too much.

While we may count this investment as non-productive, we must remember that the capital required in the tanning industry is relatively small as far as fixed assets are concerned. After all, the greatest investment that we have in our business is hides, and the more often we can turn this capital, the less of it we need, but it still remains the biggest part of our investment.

In some cases we may be able to realize a little revenue from what we can reclaim in a sewage disposal plant. However, before we bank too much on this we will have to consider the cost of this reclamation. It may cost far more to reclaim and market this offal than it is worth, especially in this day of high wages, but it is a point that should not be overlooked.

Throughout these years of Government subsidies and what have you, the tanning industry has done a pretty good job of standing on its own feet, and I think that with a realistic approach to this question each one in his own particular way can work out the solution and still not forget that we are in business for profit.

In order to achieve this it is necessary to manage well, make a good product, market it at a profit and build up good public as well as business relations.

We must, we can, and we will have clean streams.

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SYMPOSIUM ON STREAM POLUTION*

H. T. REUNING, *Chairman*

We have four papers to be presented, dealing with various phases of this whole problem. Our thought was to run through those in sequence. We are hopeful that there will be discussion and comment on these papers and on the whole subject. Let us have that after the formal part of the program.

Your Committee has been concerned with the problem of stream polution for many years. It was organized in 1938, I believe, and we have tried to treat the subject as best we could within the limitations of the Committee. In doing that, we published several articles which we are hopeful were of interest. We have now reached the point where the situation is serious. A good many of us heard Ed Thorstensen this morning. He brought out a lot of things we don't like to hear.

We hope to stay in business, and in order to comply with requirements of the various control agencies, we certainly are going to have to do something about the situation.

Our symposium has a two-fold purpose: first, to impress upon the industry the important and serious necessity of the problem; second, to outline procedures for controlling waste. In the general discussion of the matter, something may come up that will help someone with his problem.

We all know that in the manufacture of leather, different processes may be employed. There are two main definitions. One is vegetable and the other is chrome. We have asked Mr. J. W. Harnly, a member of our Stream Pollution Sub-Committee, to discuss the subject of chrome waste. The vegetable tanning process requires different treatment for waste recovery. F. F. Marshall will discuss the "Treatment of Vegetable Wastes". Several years ago, a new tannery was built in Tennessee, wherein cognizance was taken of the importance of waste treatment to prevent stream pollution. F. L. Collins, will tell us of the treatment plant at Bolivar, Tennessee.

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The Tanners' Role in the Water Pollution Control Programs*

By HOWARD T. REUNING

Keystone Tanning and Glue Co.,
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Although the necessity for correction and abatement of stream pollution has long been apparent, it has only been in recent years that an effort has been made to attack the problem on anything approaching a nation-wide basis.

Prior to World War II, a number of the States had enacted anti-pollution legislation and were preparing to embark on an intensive program to not only prevent new pollution but also restore the streams to at least a reasonable degree of cleanliness. These activities necessarily were suspended during the war years but have been resumed with increased vigor upon cessation of hostilities.

Legislation dealing with water pollution now is in effect in nearly every State. The problem of control of many interstate waters is being handled on a cooperative basis through formal agreements and compacts. A Federal Water Pollution Control Act (Public Law 845) was enacted by the 80th Congress and became effective upon approval by the President on June 30, 1949.

Streams and other bodies of water have been used for generations as a medium for disposal of both domestic sewage and the process wastes of industry. At first, the relatively minor volumes of these materials were quite readily assimilated. With the rapid growth and concentration of residential and industrial areas, however, the natural assimilative capacity of the streams soon became inadequate to prevent despoilment of the waters. Consequently, many of our streams today are grossly contaminated and public water supplies are endangered, aquatic life has been destroyed, and the water impaired for most industrial purposes.

The primary objective of present efforts in pollution abatement is the conservation of our water resources for the protection of the public health and for the future economic well-being of the nation. The ultimate goal is not the restoration of pristine purity but rather a judicious utilization of these resources for the needs of a modern civilization.

The three principal sources of pollution are erosion, domestic sewage and industrial wastes. Each source affects a stream in a different manner but individually or collectively can render the water unfit for further use without expensive treatment. Industrial wastes generally are considered to constitute not only the greatest source of pollution but also to present the most difficult problem of correction.

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The water pollution control statutes of the several States, although differing materially in requirements and administrative procedure, are based on the common law which sustains the right of a riparian owner to have the water flowing past his property unimpaired in quality. In many of the laws, pollution has been defined and special boards established to promulgate the necessary regulations for preservation of stream quality.

The stream control law in Pennsylvania is recognized as one of the most comprehensive in the country. Inasmuch as this law has been used as a model for similar legislation in a number of States, a summary of its provisions and the procedure being followed particularly with respect to industrial wastes, will be of interest.

Pollution is defined as "noxious and deleterious substances rendering unclean the waters of the Commonwealth to the extent of being harmful or inimical to the public health, or to animal or aquatic life, or to the use of such waters for domestic water supply, or industrial purposes, or for recreation". The Sanitary Water Board is empowered to determine when the discharge of any industrial waste constitutes pollution in accordance with this definition, and to establish standards "whereby and wherefrom, so far as reasonably practicable and possible, it can be ascertained whether any such discharge does or does not constitute pollution as defined."

After careful study, the streams of the Commonwealth were classified and minimum degrees of treatment assigned. A number of public hearings were held to explain the purposes of the program and the requirements involved. All sewage was to be treated "at least to a primary degree", with additional treatment as required in certain localities. Industrial wastes were to be given an equivalent degree of treatment.

The Board subsequently began the issuance of notices to all municipalities and industries to prepare plans for compliance with its orders, the notices specifying that the plans must be submitted for approval within approximately one year.

The notices sent to industrial establishments where primary treatment was required contain the following:

> "As a gauge of the degree of treatment required of your wastes, primary treatment of municipal sewage is construed to be such as, in the opinion of the Sanitary Water Board, will remove at least 35 per cent of the organic pollution load as measured by the biochemical oxygen demand test; will provide effective disinfection to control disease producing germs; will provide satisfactory disposal of sludge; and will produce a final effluent that is suitable for discharge into the receiving stream.
>
> As applied to your wastes, please note that this last requirement necessitates the removal of oils, greases, acids, alkalis, and toxic, putrescible, taste and odor producing, and other substances inimical to the public interest in the receiving stream".

Where additional treatment is required, it is necessary to remove practically all of the suspended solids and at least 85 per cent of the biochemical oxygen demand.

Following approval by the Board of plans for the required treatment facilities, a permit is issued authorizing the construction and operation of these works. In the issuance of this permit, neither the Commonwealth nor the Board "assumes any responsibility for the feasibility of the plans or the efficiency of the operation of the plant to be constructed thereunder."

Due to the confused situation in the construction field, the Board at first did not press for a definite installation schedule. Last year, however, all municipalities and industries in the Delaware and Schuylkill River Basins were notified the construction of the required treatment works must be completed within approximately two years. It may be anticipated that similar orders will be issued for the balance of the watersheds in the Commonwealth within the near future.

The enactment of Public Law 845 marked the successful conclusion of nearly fifty years effort to obtain legislation dealing with stream pollution on a nation-wide basis. The extent of Federal participation in this program is stated clearly in Sec. 1 of the Act; "it is hereby declared to be the policy of Congress to recognize, preserve and protect the primary responsibilities and rights of the States in controlling water pollution; to support and aid technical research to devise and perfect methods of treatment of industrial wastes which are not susceptible to known effective methods of treatment; and to provide Federal technical services to State and interstate agencies and to industries, and financial aid to State and interstate agencies and to municipalities, in the formulation and execution of their stream abatement programs."

The Act further provides for the encouragement of uniform state laws and cooperative activity between States for the prevention and abatement of water pollution. Responsibility for administration is assigned to the Public Health Service and the General Services Administration.

A Water Pollution Control Board is established to review the policies and program of the Public Health Service in carrying out its functions under the Act and for the purpose of making recommendations to the Surgeon General. Personnel of the Board shall include a representative of industry among the six public members.

The Act also provides that the Surgeon General shall "prepare or adopt comprehensive programs for eliminating or reducing the pollution of interstate waters and tributaries thereof". If pollution of these waters is found to endanger the health or welfare of persons in a State in which the pollution does not originate, both the offender and the control agency of his State are to be notified. This notification may outline remedial measures and specify a "reasonable time" for compliance. In the event abatement of the nuisance is not begun in accordance with the Surgeon General's recommendations, a

second notice is to be sent to both parties and the agency advised that suit may be instituted against the offender. A suit, however, cannot be entered by the Attorney General without a prior public hearing and then only with consent of the control agency of the State in which the offender is located.

Although the problems of industrial waste treatment are considered to be a responsibility of the industries themselves, the Act authorizes the appropriation of funds to finance research that will lead to the development of treatment processes for wastes that presently are not susceptible to recognized methods of treatment. An industry may, through its local State agency, request that the Surgeon General "conduct investigations and research and make surveys concerning any specific problem confronting any. . . . industrial plant, with a view of recommending solution of such problem."

A program for administration of this Act, which became effective on June 30, 1948, is being rapidly formulated. Early in 1949, members of the Water Pollution Control Advisory Board were appointed. The Board recently adopted a proposal for establishment of a National Technical Service Task Committee on Industrial Wastes. This group will serve as a coordinating agency between industry and the Public Health Service on the industrial waste problem.

In order that Federal activities may be coordinated properly with those of the State and interstate agencies, ten of fourteen proposed river basin offices have been established and are now functioning. These offices are working with the control boards of the several States in the preparation of comprehensive programs for stream improvement. In developing these programs, the abatement measures necessary to protect the streams as a source of public water supply, for propagation of aquatic life, and for industrial and recreational purposes, will be recommended.

As a result of the stimulation and cooperation provided at the Federal level, it may be anticipated that the several States will not only accelerate their efforts toward the enforcement of existing legislation dealing with pollution abatement, but also extend their authority to deal with the matter on a more effective basis.

The problem of industrial pollution is serious. Trade wastes have been estimated to be equivalent to the raw sewage of approximately 60 million people. Unfortunately, industry in the past frequently has failed to accept its full responsibility in the matter of waste treatment. This attitude has contributed in a large measure to the gross pollution of many of our streams, a condition which today reacts detrimentally both to industry itself and others entitled to share in the use of public waters.

The correction of this problem is being accorded high priority by all control agencies. Methods for treatment of the wastes discharged by most types of industry are known, and installation of the required facilities in these cases is expected to proceed without undue delay. Research projects are being

established to develop treatment procedures for the relatively few wastes that cannot at present be handled in an economically feasible manner.

The tanning industry recognized a number of years ago that the wastes produced in the manufacture of leather were of a highly polluting nature. Individual companies, and segments of the industry working cooperatively with governmental agencies, conducted laboratory and pilot plant experiments on methods for treatment of these wastes. As a consequence of this research, processes for reduction of most of the objectionable characteristics of tannery wastes were devised. These procedures are considered to be practical and capable of producing an effluent that can be assimilated readily by a stream.

A number of tanneries have installed treatment facilities employing the principles developed during these investigations, thereby enabling these plants to comply with requirements of the local control agency. Other papers this evening will review these principles and their application in the treatment of both crome and vegetable wastes.

The solution to a specific problem in tannery waste disposal is a matter that necessitates a detailed study of a number of factors. The degree of treatment that must be provided is dependent upon the regulations of the control agency for that locality. The volume of wastes and availability of land are other factors that will have an important bearing on the design of the works to be required.

In preparation for the eventuality of waste treatment, it is advisable that a tannery proceed immediately with a comprehensive survey of its problem. The voluntary initiation of a study of this nature may be interpreted in some quarters as admission of the existence of a disposal problem, with the result that the control agency may press for installation of remedial measures. This will not be the case, however, and a survey now will afford an opportunity to review the problem thoroughly prior to the receipt of notification to abate the nuisance.

Considerable publicity has been given to the profits that waste treatment installations have returned. Unfortunately, the tanning industry cannot look forward to an additional item of revenue from this source. There is, however, an opportunity to effect a substantial saving in capital and operating costs through waste prevention measures. The expenditure that will be required for pumps, settling tanks and other equipment is proportional to the volume of wastes that must be handled. In many tanneries, water consumption is greatly in excess of the quantity actually required in the process, and a definite reduction frequently is possible through ordinary good housekeeping practices. A study of the practicability of segregation of the cleaner wash waters, for recirculation or discharge direct to the stream, may disclose where additional waste volume reductions may be effected.

The design of the treatment facilities should be adequate for present requirements, with provisions for additions that may become necessary as a consequence of future plant expansion. A wide range of standard sewage plant equipment, adaptable in the handling of tannery waste, is available. The type selected, however, should be of relatively simple design for best results.

Waste treatment is a "must" that industry no longer can ignore. Economically feasible methods for the abatement of pollution caused by most industrial wastes are known and installation of the necessary disposal facilities are being required. Although regulatory legislation has been provided to deal with the problem on a firm basis, it is evident that punitive measures will not be applied unless industry refuses to cooperate in a reasonable manner. The despoilment of our water resources, attributable in a considerable degree to the use of streams as a convenient dumping place for industrial wastes, can no longer be tolerated. The public is demanding, and industry needs, clean streams.

Treatment and Recovery of Chrome Wastes*

J. W. Harnly

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There are several principle approaches to this problem, namely: 1. Obtain 100 per cent take-up of chromium from the tanning liquors, which would automatically eliminate the problem. While this approach may not be feasable as a complete answer, any progress in increasing the take-up will tend to mitigate the problem. 2. The opposite to this would be to use chromium freely, but to recover all that remains in the waste liquor. 3. Reuse through the replenishment method, that is reusing any chromium remaining in the spent tan liquor. This is accomplished by adding the required amount of chromium and other materials that have been reduced and then building up to the original volume. This method is complicated by the build up of semi-inert materials such as calcium or sodium sulfate and by the presence of contaminants, such as fleshings or soluble protein degradation products.

Then we can ask some practical questions. In most cases the presence of chromium in a tannery waste effluent is not objectionable *per se*. It is a very small portion of the total or even of the suspended solids. It is insoluble, imparts no color and is nontoxic, since, due to the normal alkalinity in most tannery liquors, it is completely precipitated as chromium hydroxide. So let us assume that we do not have to remove the chromium from our effluent. Then can a reuse or recovery system, be made to stand on its own feet?

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However, we know from past experience that chromium can become a scarce material of high priority and that because we have an insufficient amount available, we must get 100 per cent use of what we have. Then under these conditions, which of the above methods appears to give the greatest promise or may it be a combination of some of the above or is some other approach advisable? These are some of the things we would like to have considered in the discussion to come.

N. C. Benrud will discuss one phase of the recovery of chrome, J. W. Harnly will outline a recovery system, while W. A. Morris will discuss the merits of these approaches.

RECOVERY PROGRAM*

By J. W. Harnly

This is a discription of a recovery system that was used during the last war. Approximately 125,000 pounds of Cr_2O_3 were available for recovery yearly. This would be equivalent to better than ¼ million pounds of bichromate or more than ½ million pounds of tanning material such as Koreon or Tanolin R. This material was in 15 to 20 thousand gallons of exhausted chromium liquor daily which was collected in a central sump. It was pumped to settling basins with caustic soda, fed into the suction of the pump, in an amount to maintain a pH of approximately 8.0.

On settling this gave a chromium-free supernatent liquor which was decanted. Then more exhaust liquor was added and again decanted. This was continued until a volume of slurry was obtained that represented five volumes of waste liquor. This slurry was then washed by adding fresh water, agitating, settling and decanting. After several washings to bring the soluble salts down to a maximum control value, the slurry was allowed to stand until ready for use at which times the supernatent was again decanted. At this stage one volume of slurry represented 10 volumes of waste liquor.

This slurry with its known chrome content was then used in place of water in making up chrome stock liquors. The stoichiometric requirement of sulfuric acid was added and the dry, fresh chrome tanning material, either a prepared tan or bichromate, was also added. In both cases on either agitation or reduction the chromium hydroxide readily dissolved giving a stock liquor of the desired basicity and concentration.

Several systems for further concentration of the slurry such as the use of a Dorr thickener or a Sharpless G-2 autojector or a filter press were devised, but since the concentration already obtained was sufficient to allow the use of all the chrome recovered, no active work was done on further concentration except a few laboratory experiments.

This sytem involved no problems with build up of undesirable components since they were removed in the washing process. Also the system could be

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made to show a profit since chemicals, water, and labor cost less than the value of the recovered chrome. However, due to other costs such as maintainance, over-head and depreciation it was not the type of venture that would interest profit-seeking capital.

Treatment and Recovery of Chrome Wastes*

By N. C. Benrud

I will tell briefly of the reuse of chrome liquors in paddle tanning. Paddle tannage, because of the large volume necessary to float the stock, was a wasteful method of tannage. I say, was wasteful, because we now think we are tanning economically. During war time, the scarcity of chrome made it necessary to save all that was possible. Therefore, Mr. Thorstensen, together with Mr. Serfass, worked out a method of conservation suitable for this type of tannage. All residual tan and salt had always been drained to the sewer and fresh paddles started for each pack of leather.

We now simultaneously pickle and tan. After neutralizing and a period of running to allow equilibrium to be established, the sides are hauled out. The volume is corrected and a sample is analyzed for salt, acid, and chromic oxide content. With this information the tan foreman determines the amount of salt, acid and tan liquor necessary for the next lot of hides. Tanning proceeds in the same liquor. We tan seven consecutive lots of hides in this manner. The liquor is then used to retan splits. After the splits are hauled out the remaining liquor goes to the sewer. This liquor will analyze approximately 14 pounds of chromic oxide per paddle. At this rate we are running into the sewer two pounds chrome oxide per 3300 pounds white weight stock with no loss in retanning of splits. Salt run into the sewer is only about 70 pounds per pack. Summing it up from the pollution angle, our tan yard wastes for each 1000 pounds of raw stock approximately 54 gallons water, 0.6 pounds chromic oxide, 22 lbs. of salt and a very small amount of sulfuric acid.

We make no attempt to recover retanning wastes other than maintaining economical operating conditions.

CONSERVATION OF CHROME*

By W. A. Morris

In consideration of the foregoing observations as to the importance of conserving chrome tanning material we may regard the matter of efficient take-up of chrome from the tanning liquor as a first step. I am speaking of the drum tannage with which I am more familiar than paddle tannage.

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What percentage of chrome take-up should we expect to achieve from the original tanning liquor?

I believe it is reasonable to assume we can have an 85 per cent take-up, approximately, on side leather in the drum tannage. I do not know what the average experience may be in this direction; however, an 85 per cent utilization of chrome can be accomplished by proper adjustments in the tanning procedure. In some calf-skin tannages the amount of chrome remaining in the waste tanning liquor may be somewhat greater.

As a second step, two procedures have been described previously this evening for recovery of the greatest amount of the remaining chrome content from waste tanning liquors. There are undoubtedly some tanneries in which the plant lay-out, or existing facilities, may not be such as to permit the sort of procedure of recovery which has been described by Mr. Harnly. In such instances, and where it may not be economically feasible nor possible due to plant lay-out, another means may be available. It should not be too difficult to collect the waste liquor from the tanning drum and, by suitable installation of sump or tanks, to pump this liquor over for re-use, either to be used as a sort of pre-tannage, or to be fortified with fresh chrome liquor in tanning subsequent loads of skins or sides.

Treatment of Vegetable Wastes*

By F. F. Marshall

Keystone Tanning and Glue Co.
Ridgway, Pa.

The right to move various sewages to the sea through natural waterways must be defended by the public and industries in the legislatures of our states. Nature herself uses the streams to remove wastes from the hills and mountains. Anyone who has studied streams will find heavy banks of rotted leaves, wood, etc., which decay and form organic pollutions. In the flood seasons, like some of our industrial brethren, nature flushes these wastes onward toward the sea. Were it not so, our pure streams would eventually become stinking swamps, and ponds, and would be cleaned by fermentation during the hot months.

This attitude may appear as a reversal of that normally coming from one who has held high office as a conservationist, but in no wise indicates a desire to use our streams as the dumping grounds for unnecessary waste and filth. It is time to clean up, and those among us who have delayed should pay the penalty for their neglect without complaint. For they in turn have penalized the others who are meeting their obligations.

* Forty Sixth Annual Meeting, French Lick, Ind., May 31, 1950.

In our own industry the know-how of treating vegetable tanning wastes has been found and tested and broadcast to the entire industry through your Journal and through trade papers. The most comprehensive study of this subject made to date was that of the Tannery Waste Disposal Committee of Pennsylvania, in a condensed report to the Sanitary Water Board on the Treatment of Tannery Wastes, [*J. Am. Leather Chemists Assoc.*, **26**, 70 (1931).]

Each tanner has an individual problem depending on the size of the plant in relation to the size of the receiving stream or in case of the city tannery, the size of the available disposal plants. Each class of tannery has been studied by Mr. Reuning's committee, and others and treatment plans are established.

I read from an article in Public Works Magazine, March, 1947 entitled, "An Effective Tannery Waste Treatment Plant", by H. T. Reuning and R. F. Coltart, describing the waste treatment works of the Keystone Tanning & Glue Co., Inc. at Ridgway, Pa.

Flow.—Waste waters from the beam house, scrub house, and finishing department are collected in two sumps. Approximately 85 per cent of the total flow is from the beam house and the pH of the combined wastes varies from 11.0 to 12.0. Two 3-inch centrifugal pumps, operating eight hours per day, pump the wastes to the treatment plant. The pumps are float controlled and the average flow to the treatment plant is 35,000 gallons per hour. During peak operation the maximum pumping rate is 50,000 gallons per hour.

Design.—The treatment plant is designed for mechanical flocculation of these wastes, sedimentation in a mechanically cleaned settling tank, flow equalization for uniform discharge to the receiving stream and mechanical sludge dewatering and handling facilities. The spent tan liquors are evaporated for recovery of salable by-products.

On entering the treatment plant the wastes are flocculated in a mixing tank 16′0″ wide x 13′0″ water depth x 33′0″ long. At the average flow rate of 35,000 gallons per hour a theoretical detention period of 1.5 hours is provided. The mechanical mixer consists of three sets of redwood paddles mounted on a horizontal shaft arranged parallel to the liquid flow through the tank. The peripheral speed of the paddles is proportioned for maximum flocculation of the fine suspended solids. A processed malleable iron chain drive connects the paddle shaft to the motorized helical speed reducer mounted on top of the tank.

After flocculation, the waste passes to a settling tank 33′0″ wide x 13′0″ average water depth x 70′0″ long. This tank is equipped with two longitudinal and one cross sludge collector for moving the settled solids into a hopper in one corner of the tank. Each conveyor consists of a double strand of 6″ pitch processed malleable iron chain operating over three sets of sprockets. Redwood flights are spaced on ten foot centers on the longitudinal collector

and on 5′0″ centers on the cross collector. The conveyors operate at a speed of about 1 foot per minute. One special motorized reducer with three output shafts is arranged to drive the three collectors. The average theoretical detention period is 6.5 hours.

The effluent from the settling tank passes to a flow equalization basin with a storage capacity of 370,000 gallons. Two swing-joint float-supported effluent pipes are used to withdraw the treated waste from the basin through a float-controlled valve and orifice box at a uniform rate throughout a 24-hour day.

Three 40-inch solid wall, basket type, suspended centrifugals are provided for dewatering the settling tank sludge. Each one is powered by a 15 HP direct-connected, 1200 rpm. motor.

Operation.—The various wastes which are discharged intermittently are thoroughly mixed and flocculated in the mixing tank. The mechanical mixer is run continuously throughout the period the tannery is in operation.

Due to the fact that it requires 16 hours per day to centrifuge the sludge, the sludge collectors must be operated for this period each day. During normal operation a sludge blanket about 12 inches deep is maintained in the bottom of the settling tank to permit as heavy a solids concentration in the sludge as possible. The sludge is withdrawn through an adjustable decanting pipe into a storage sump from which it is pumped to a flow distribution box. From this box the flow is delivered to each of the sludge dewatering machines at a rate of 15 gallons per minute. A complete operating cycle requires sixty minutes. The sludge from the centrifugals is elevated to a watertight dump truck outside of the building.

Results.—The suspended solids of the plant influent average 2,500 parts per million. An average of 250 parts per million suspended solids is found in the final effluent discharged to the river. This represents 90 per cent reduction, but on the basis of 24-hour discharge, a reduction of 96 per cent in suspended solids loading on the stream is obtained.

The five day Biochemical Oxygen Demand of the plant effluent averages about 350 parts per million. The actual tannery discharge handled in the treatment plant contains approximately 600 parts per million BOD, but if the spent tan liquors were taken into consideration this would amount to about 1200 parts per million.

If credit is taken for the elimination of spent tan liquors by evaporation and a 24-hour uniform instead of 8-hour intermittent discharge, the total 5-day BOD reduction can be considered at 90 per cent.

The centrifugals discharge about 2500 gallons of 85 per cent moisture sludge per day. This represents about 10 per cent of the volume of sludge received from the settling tanks. The sludge is disposed of by dumping in a low area on the tannery property. It makes excellent fill and no objectionable

conditions are created. The discharge from the centrifugals is mixed with the untreated wastes and is resettled in the settling tank.

Some tanneries are fortunate in having large available areas for impounding their wastes. Treatment here will depend upon the size and type of the receiving stream and the degree of treatment required by existing regulations. Mr. Reuning describes such treatment in a paper published in *Sewage Works Journal*, **20**, 525 (1948) entitled "Disposal of Vegetable Tanning Wastes", part of which I will read.

Many tanneries provide sufficient lagooned areas to allow for routine storage of all liquor and release to the stream only at controlled rates during high stages. The addional dilution available at these times permits of harmless assimilation.

At the average tannery it will be necessary to pump the wastes to the treatment plant. A collecting sump, situated where all wastes can be delivered to it by gravity, should be provided. Horizontal or vertical centrifugal sewage trash pumps, float controlled for automatic operation, are relatively trouble free. The installation of two pumps, each capable of handling the average daily volume, will insure uninterrupted operation. Screening of the raw wastes generally is effective for removal of rope and the heavier solids in the raw wastes that frequently tend to clog the pumps. Rotary drum-type screens have been found quite satisfactory for this purpose and should be installed in the main sewer at the sump.

Units for primary sedimentation may consist of earthen basins or tanks constructed of concrete, wood, or steel, and operated on either the "flowing-through" or "fill-and-draw" principle. The efficiency of either method in removal of settleable material is quite comparable under similar design conditions. The use of mechanical equipment to facilitate mixing of the wastes and removal of sludge has been found practicable at a number of plants.

In an installation of this type, the wastes would be pumped first to a mixing tank having a capacity equal to the maximum hourly inflow and equipped with paddle mixers operating at a slow peripheral speed. The mixed wastes would flow to a settling tank designed to provide a minimum theoretical detention period of 6 to 8 hours. The wastes should be introduced through submerged inlets distributed across the end of the tank. The effluent weir should extend the full width of the opposite end of the tank. The sludge collecting mechanism can be of standard sewage plant design but should provide for longitudinal and cross collection to a single withdrawal point. The sludge will flow readily under a low hydrostatic head or can be removed with centrifugal pumps.

In "fill-and-draw" primary sedimentation, a minimum of two units should be provided, each unit to have a capacity equal to the total daily waste volume, plus 50 per cent for sludge storage. Horizontal, float-supported, perforated pipes can be used to withdraw the settled effluent. The units

should be located so that the sludge will flow by gravity to the outlets with a minimum of hand labor. The sludge must be removed at regular intervals to maintain good efficiency.

The effluent from either method of sedimentation should be resettled prior to discharge to a stream. A single tank or basin, with a capacity equal to one and one-half times the daily waste volume, can serve as a combined resettlement and flow equalization unit. The final effluent should be withdrawn through float-supported collector pipes to a control box equipped with a quick-acting valve. The discharge to the stream should be at a uniform rate over the 24-hour period.

The disposal of sludge is a difficult and expensive problem at any tannery. The average daily quantity amounts to approximately 8 per cent of the waste volume, with a moisture content ranging from 95 to 98 per cent. Where ample land area is available the sludge may be stored indefinitely in large lagoons. In semirestricted areas, shallow drying beds are practicable. A series of beds, to provide approximately 2 sq. ft. of surface area for each pound of raw hide processed, would be required. The beds should be used in rotation and top water removed to facilitate drying. The sludge will dry readily to 80 or 85 per cent moisture content and can be removed satisfactorily with a power shovel and trucks. Centrifugal dewatering will reduce the sludge volume from 80 to 90 per cent and is a practical, although expensive, solution for those plants located in closely builtup sections.

The individual nature of any problem in tannery waste disposal precludes more than a brief outline of the general measures adaptable in the treatment of the process waters. A thorough study of all phases of the problem is essential to the design of a treatment works that will produce an effluent which can be assimilated harmlessly by the stream.

Mr. Reuning and members of his committee are most willing to assist you in your problem.

Tannery Waste Disposal at Bolivar, Tennessee*

F. L. Collins

International Shoe Co.
St. Louis, Mo.

Most of you have probably heard or read Mr. Richards' description of our Bolivar Tannery and some of you have seen the plant. However, in review, Bolivar is located 70 miles east of Memphis (which has nothing to do with sewage disposal) on the Hatchie River (which is important). It is about

* Forty Sixth Annual Meeting, French Lick, Ind., May 31, 1950.

125 miles "down the Hatchie" to the Mississippi. Some 200 people are employed to process about 60 thousand pounds per day of green salted kip and cattle hides into chrome tanned upper leather.

The plant site of 175 acres is outside the city limits and northeast of the town. This is presumably "downwind" from town if the prevailing winds prevail. Shortly after opening the plant, the wind blew southeast for 15 days straight.

The storm water, of course, is kept separate from the process water. The plant area is drained directly to the Hatchie through a combination of natural and man-made ditches. The disposal of the water from the 125 thousand square feet of roof is somewhat unique in that no eave-gutters or downspouts are used except on the flat-topped administration building and refrigerated hide storage. A "V"-shaped concrete gutter at ground level receives the roof water and transports it to one of the drainage ditches.

The laws of the State of Tennessee require that any new plant submit engineering plans of collecting sewers and treatment works in advance of operation. This law was designed to protect small plants and municipalities from "shysters" and causes no trouble or delay when reputable engineers are on the job. Estimates of volume and concentration are also required, prior to operation. The State either has on file, or will obtain, data on the ability or suitability of the receiving stream to carry the proposed waste and a temporary permit is either issued or denied based on the available data. There are no hard and fast rules, which is as it should be. If the waste can be shown to be free of toxic chemicals and pathogenic bacteria, reasonably neutral, free of floating solids, and does not deplete the dissolved oxygen to a point dangerous to aquatic life or likely to cause septic condition either by itself or due to an additive effect from nearby plants or communities, then a temporary permit will be issued. After this temporary permit is issued, the plant may be put in operation and shortly thereafter, the State sanitary engineers move in with a completely staffed portable laboratory for a survey of the plant effluent and the condition of the stream above and below the plant If this survey is satisfactory, a permit to operate, as long as there are no changes in conditions, is granted. Any plant expansion or radical process change will require a re-survey.

The state does not demand but strongly recommends that periodic estimates of volume and analysis of the effluent and receiving stream be made. This information, together with occasional spot checks by the State, is primarily for self-protection in case of subsequent law suits. It is my opinion that once a plant has been granted a permit and is operated in a reasonable manner, then a second plant will not be allowed to build close enough that the additive wastes from the two plants will overload the receiving stream. I consider the policy of the State of Tennessee to be reasonable and farsighted and in the hands of capable personnel.

Based on previous experience at our Wood River Tannery, we submitted preliminary plans and data to the State of Tennessee and a temporary permit was issued. After the plant was in operation, a complete survey was made by the State and an operating permit issued.

The collecting sewer system is composed of open concrete channels covered in the working areas with removable iron grates. This permits easy cleaning and some aeration. The channels are from 6 inches to eighteen inches wide and from 6 inches to several feet in depth. The slope varies from 0.3 per cent to 1.25 per cent. It was intended to have as high a velocity as possible in the sewers to obtain as much self-scouring action as possible.

The usual practice of recovering trimmings, fleshings, and hair is followed. The rotating screens for hair removal are the only mechanical devices on the entire sewer system.

The pickle liquors are the only ones that are being recharged and reused. Collecting sumps for exhaust chrome liquors have been provided, but up to the present time have not been used.

The total mixed tannery and domestic wastes of about ¾ million gallons per day flow by gravity to one of two large earthen basins where primary sedimentation takes place. The basins are used on alternate weeks in the hope that some aeration and recovery will be obtained. No data are available on this point.

Each basin is approximately 200 by 500 by 4 feet deep but is operated at a depth of only 18 inches. The entire depth is not utilized because previous experience has shown that it is next to impossible to air-dry four feet of sludge when cleaning becomes necessary. This method of operation gives a calculated detention period of two days when the basins are clean. The detention period decreases, of course, as sludge is accumulated and data on suspended solids and biochemical oxygen demand will be used to determine when cleaning is necessary. The plant has been in operation about two years and cleaning has not been necessary to date. According to calculations, the basins should now be full of sludge to the 18 inch flow line and cleaning will be necessary some time in the near future. However, B.O.D. and suspended solids are still being satisfactorily reduced. The engineering department is investigating the possibility of dredgetype cleaning, with the sludge being pumped to waste land in the plant area and plowed under. This would eliminate the necessity of partially drying the sludge before removal. In an emergency, the flow line can be raised, up to 4 feet, to provide a longer detention time.

The large area of exposed water created an odor problem. There is some decomposition in the basins, as evidenced by gas formations, and a drop in pH to about 8. The net result is the evolution of hydrogen sulfide. It has been necessary to add a thousand pounds per day of "Ferri-Floc," a hydrated ferric sulfate, for odor reduction. Through the use of this material we are, temporarily at least, back in the good graces of the community.

The basin influent is not provided with any metering device and estimates of volume at this point have not been too satisfactory. We are considering the installation of a Parshall flume for this purpose. The basin effluent flows over a built-in sharp-crested rectangular weir and here the volume estimates are considered reasonably accurate. The basin effluent flows about 850 feet in an 18-inch closed tile to a natural drain. This natural drain is entirely on tannery property and receives most of the surface water in addition to the backwash from the iron removal system and boiler blow-down. It is about one-fourth mile from the end of the sewer tile to the Hatchie River.

The Hatchie River has a minimum recorded flow of 125 cubic feet per second and thus we have reasonable assurance of a 100 to 1, or better, dilution ratio. Based on dissolved oxygen determinations of the river water, the effect of the tanery effluent has not been detectable up to the present time. The Hatchie is considered a good channel-catfish stream by commercial fishermen and no complaints have been received from them. In fact, a few fishermen have stated that fishing has improved since the tannery went into operation. This improvement might be expected since it has been shown that the addition of commercial fertilizer to fish pounds is definitely beneficial provided the oxygen is not depleted.

Available data indicates that the method under discussion removes 90 per cent, or better, of the suspended solids and 50 per cent of the biochemical oxygen demand. The B.O.D. of the basin effluent is about 200 parts per million and, with a volume of ¾ million gallons per day, the oxygen requirement is 1250 pounds per day. Based on 0.17 pounds of oxygen per person per day, then the load on the stream is equivalent to a population of 7000. The Hatchie River usually carries about 6 ppm. dissolved oxygen so a dilution of 33 to 1 supplies the theoretical amount of oxygen, even if no further aeration occurs.

The removal of suspended solids by primary sedimentation is relatively simple but the disposal of the accumulated sludge is a major problem. Secondary treatment is inefficient. The procedure used at Bolivar is not unique, the results are not startling and the process is not adaptable to plants that have either a shortage of land for sludge disposal or insufficient water for dilution purposes.

Discussion

A. N. Kay: I should like to ask Mr. Collins to check some of these figures here. As I remember, he said there was a minimum dilution of a hundred to one: the dissolved oxygen of a dilution of 33 to 1 satisfied the B.O.D. of the treated effluent. The river above the tannery has dissolved oxygen of 6 parts. Would that imply that the river below the tannery only has 2 parts.

Collins: This is correct. However, that is theoretical on a five-day demand. The river water analysis below the tannery still has 6 parts per million as far as two days down stream.

Since we have been in operation, the stream has never approached minimum flow. Most of the time it has been out of its banks.

JAMES F. WEBBER: I would like to ask Mr. Collins whether any septic action took place in the two-year use of your chrome liquors.

Is there not a possibility for micro-biological activity in the re-use of the chrome liquors?

COLLINS: There is a possibility. We did not observe any biological activity.

HARRY BERKENSTEIN, JR.: One of the major points of an older tannery that has been overlooked entirely is this: In most of the tanneries, the sewer systems are laid out in such a way that they either enter into a river or enter into city sewers. However, in redesigning you have to move all the effluent to a large area and this requires quite a cost in redesigning the entire sewer system. I think that is worth keeping in mind, there. That can run into a great deal of money.

REUNING: That is a good point. Most of our tanneries are not as forntuate as Mr. Collins', starting out from scratch. When these tanneries were built little thought was given to waste disposal. A hole or half a dozen holes were made in the building somewhere and through which wastes drained. We have to spend a lot of time and money now figuring how to get it around to a point where we can handle it. I don't know what we can do about it, unless we tear down the building and start all over, and that is not practicable. That points up the individual nature of the whole problem in that particular respect.

HARNLY: I just want to make a remark in line with what Mr. Berkenstein brought up.

A large tannery will use about two million gallons of water per day. There was a time when I thought that was a lot of water. However, I attended a sewage symposium in Chicago recently, where many industries were represented. A man who represented Standard Oil stated that at Whiting, Indiana, they use 185 million gallons of water a day, and that one of their principal problems was the fact that their sewage system was antiquated; that these installations had been started thirty-five years ago and were a conglomeration of open ditches and concrete sewers and brick sewers, and that most of the money had to be spent on getting the effluent together and finding out what they had. So the leather industry is not the only industry confronted with that problem.

MR. SHANNON: My point is just a minor one. I was particularly interested in the aspect of hydrogen sulfide from your settling tanks. Has the development of hydrogen sulfide been studied, and is that a factor in waste disposal as a new sanitary value?

COLLINS: It is a factor.

SHANNON: What does it develop from? Maybe we can get some preliminary thoughts.

COLLINS: We use an appreciable quantity of sulfide in the form of sodium sulfide for unhairing purposes, and under the system that we have there is evidently some decomposition of organic matter in the basin producing more hydrogen sulfide.

MARSHALL: I might add that a recent survey by a group of sanitary engineers found that 95 per cent of the sulfide coming from the tannery had been removed by the treatment described in the second part of my paper.

MR. KAY: Does any of the panel have any experience or ideas as to how much of the so-called B.O.D.—overall B.O.D.— can be removed by removing the chemical oxygen demand as distinct from the actual B.O.D.?

DR. KOPPENHOEFER: Is grease contamination merely a matter of suspended solids, or does that in any way affect the B.O.D.?

REUNING: I believe in most cases it is a matter of one of these conditions that are prohibited, such as floating grease, as distinct from dissolved materials. If you remove B.O.D. through efficient sedimentation, and have proper skimming devices, you won't have any grease problem as far as I know.

J. F. WAGONER: I might describe very briefly the treatment which is being put into effect at one of our sole leather tanneries. The volume of waste, exclusive of the tan liquors, amounts to about 168,000 gallons per day. All of the wastes are to be collected by gravity in a sump, except the bleach and tan liquors. The bleach liquors, as you know, in a vegetable tanning process are usually dumped at one particular time of the day. These bleach liquors are to be collected in a separate tank. Then on the following day, they will be permitted to flow into the sump over the period of the eight-hour collection time, thus avoiding the sudden influx of these wastes at one time. The sump is equipped with two Marlowe sludge pumps with a capacity of 300 gallons per minute, installed in such a way as to have a maximum pumping capacity of 600 gallons per minute. The tan liquors are to be collected in two large tanks. They will then go by gravity to either evaporators or a spent liquor lagoon. The spent liquor lagoon has an estimated storage space for twelve months.

The primary treatment of the waste consists of twin Link-Belt units. These have a capacity of 216,000 gallons. The detention period of the waste in the twin Link-Belt units will be eight hours. The Link-Belt will opearte at a rate of two feet per minute. The waste will be permitted to enter across the entire width of the unit through a weir and will be discharged at a uniform rate and is so constructed that the units can be operated individually. The effluent from the Link-Belt units flow by gravity to a settling basin. The settling basin is 200′ long and 75′ wide, and has a depth of 5′ at the effluent end and 7.5′ at the influent end. This is made in this way to permit settling out of the heavier solids at the deeper end of the basin. It is estimated that it will have a detention period of four to five days. It is figured that we will

have to clean the basin only once a year. The effluent from the basin will then be discharged to the stream at an uniform rate over a 24-hour period. The sludge from the Link-Belt unit will be pumped into two large tanks of approximately 76,000 gallon capacity. Each of these tanks will hold five days' sludge. While one of the tanks is being filled, the other will be allowed to settle. It will have a period of approximately four days' settling. It is estimated that one-third of the volume will be able to be withdrawn at the end of that period. The remaining sludge will then be run by gravity to a sludge lagoon, where it will be dried.

SHANNON: We have talked about lime and effluent from the beamhouse and the fatliquors, and so on. I would like to get a little more discussion on the effective removal of the dyes that are released through sewers, and in effluents. How effective are the treatments so far in removing color from your effluent?

REUNING: In the case of vegetable wastes, we do not have much success taking the color out, unless we lagoon the tank liquors or evaporate them. The latter, of course, is a pretty expensive proposition and is only done when necessary. The states in general—I think this is a fair statement—are recognizing (speaking now of vegetable tanning wastes) that color is of secondary importance. True, the laymen judges the efficiency of a treatment, or whether or not a stream is contaminated, by the color. That is the only way he has of judging it. But they are coming to realize more and more that color does not mean too much in many cases. I believe that is more of a problem in some of the specialty leathers. Perhaps Mr. Harnly has something on that. He is familiar with these leathers.

HARNLY: There may be some specialty leathers, where it is a problem, but to my knowledge, in the ordinary run of side upper leather tanneries, the color imparted by the dyes used is not a factor at all. In fact, we found on investigation that the principal source of color was from washing the finishing machines and not from the coloring at all. This color may be concentrated momentarily, but if it goes into a collection basin, the final amount of color that is in the effluent, particularly after it has been acted upon by the alkalinity of the lime is actually negligible. The chief source of color in normal tannery effluent is vegetable tannin.

MR. WEBBER: I would like to ask whether any attempt has been made to recover by-products from tannery waste?

REUNING: I guess there is not much we can get out of it. I think there is an avenue that has not been thoroughly explored—speaking again now of vegetable wastes—and that is the tannins that we now throw out in the tan liquors. Surely there are some uses we can find for it.

I think the best thing we can do is to start in the plant and work on prevention. We have not given much thought to that. As long as we had a big enough supply of water and a large enough stream, we kept on using it. There are all

kinds of figures on how many thousands of gallons will be wasted in a month from a dripping faucet. It is astounding. We can do a lot that we have not done in the past to economize in the use of water, resulting in lower costs for the installation and operation of waste treatment methods.

MR. SCHULTZ: I would like to ask if anything has been done on the use of a trickling filter in the treatment of effluents?

REUNING: At Emporium, Pennsylvania, during the investigations by the Tannery Waste Disposal Committee of Pennsylvania, experiments were conducted on trickling filtration. The waste first was given plain sedimentation. The effluent then was treated with sulphuric acid and aluminum sulfate, and allowed to settle. The settled effluent was applied to the trickling filter. The particular filter used was, I think, a six-section affair, and the test showed no bacterial action at all. It was simply an oxidation device. To the best of my knowledge, no practical application has been made to date.

I might add that the improvement in B.O.D. was relatively minor—about 10 per cent, as I recall, and not considered commensurate with the cost of the installation.

PERRY SMITH: How much effect would simple aeration have on tannery waste?

REUNING: Very little, as I stated in the case of the so-called trickling filter at Emporium.

SCHULTZ: Some recent work has been done on brewers' effluent, in which the pH was adjusted before aeration. There was a material reduction in B.O.D. which amounted to about 80 per cent.

KOPPENHOEFER: Has there been any work done on the use of flocculents for concentrating the sludge?

REUXING: Only experimentally, as far as I know, and there again it has been considered too expensive. Sludge is the big problem in tannery waste treatment. In the plant Mr. Marshall described, concentration was effected by centrifugation. It is expensive, but does the work and is applicable where ground area is limited for the storage of sludge for a temporary or indefinite period.

RAYMOND L. MOORE: Has the use of oxygen producing vegetation been of any value?

REUNING: Referring now to vegetation that grows in streams?

MOORE: In the collecting pools.

REUNING: Not as far as I know.

WEBBER: What is the fat content of waste—the grease content?

REUNING: Speaking now of the raw waste or treated effluent?

WEBBER: The raw waste.

REUNING: I don't know that that has been measured. As I said a while ago, it comes out pretty well in sedimentation and you can use skimming devices.

HARNLY: Depends on the individual tannery.

REUNING: Your Committee has tried to be of assistance to the industry. We cannot act as consulting engineers, but maybe we can help you with the general nature of the problem.

The government, through the Public Health Service, published a booklet entitled "An Industrial Guide to the Tanning Industry". It is also known as "Appendix XI of Supplement "D" to the Final Report to the Ohio River Committee." This is available through the Superintendent of Documents, Washington, D. C. It has not been published in our Journal. Dr. O'Flaherty has run off a number of copies and we have them here. They are available for anyone interested. They contain a lot of general information that will be helpful in the solution of specific problems.

A Report on the New England Industrial Wastes Conference

June 26–28, 1950

By HOWARD T. REUNING, *Chairman*

The American Leather Chemists Association
Stream Pollution Committee

The matter of control and abatement of stream pollution resulting from industrial wastes was thoroughly reviewed during a three day meeting at Massachusetts Institute of Technology, Cambridge, Mass. on June 26-28. Sponsored by the New England Council, the New England Sewage Works Association and the Institute, the conference was attended by over 200 representatives of industry, state and federal control agencies, research groups and consulting engineers.

As a delegate sponsored by the American Leather Chemists Association the author attended this conference. For the benefit of the leather and tanning industry of the United States this report is made available. The American Leather Chemists Association have a standing committee on Industrial Waste Disposal. The committee, whose chairman is the author, seeks to bring to the attention of the industry all recent developments in the field of industrial waste disposal and stream pollution abatement.

The continued trend toward progressive degradation of many streams in the New England area, despite the existence of regulatory legislation and the efforts of control agencies, presents an increasingly serious problem. Public

water supplies have been jeopardized in some localities, recreational values impaired or entirely destroyed, and frequently expensive pre-treatment is required before the water in many streams can be utilized for industrial purposes. The sponsors of this conference were cognizant not only of the vital necessity for preservation of the area's water resources but also were aware of the numerous problems confronting both industry and the states in attainment of this objective.

Industrial pollution in New England is widespread and varies considerably in both quantity and composition as a result of the large diversification of industry. The important pulp and paper, textile, leather, food processing and metallurgical industries discharge tremendous volumes of particularly obnoxious wastes. The treatment of these wastes to prevent contamination of the receiving waters presents serious difficulties that must be resolved as a part of any overall program of pollution abatement.

The conference agenda was confined to consideration of the problems of these industries in providing adequate waste disposal facilities and to discussion of the activities of the regulatory agencies in administration of the stream control statutes. Of particular interest to the leather industry was the paper by Thomas R. Camp, Consulting Engineer, Boston, Mass. on "The Disposal of Tannery Wastes". *Mr. Camp's paper will be found very helpful in the approach to a specific problem of this nature.**

It was clearly evident from the opinions expressed by the various speakers that economic considerations frequently have prevented wider adoption of wastes treatment facilities. Technical processes for handling the numerous types of wastes in the area are available, but in many instances, industry feels that it cannot afford the additional financial burden required for these installations.

The several states in New England now have the necessary legislation to deal with industrial pollution on a firm basis. The provisions of each state law vary considerably but in general define pollution and empower the Department of Health or a specially designated agency to adopt regulations for the improvement and protection of surface waters. Failure upon the part of an offender to comply with the established requirements can lead to the application of penalties.

As a consequence of enactment of the Federal Water Pollution Control Act (P.L. 845), efforts to abate pollution have been greatly accelerated. Under this Act, protection of the public waters remains a primary responsibility of the several states. The United States Public Health Service, however, is authorized to proceed with the development of comprehensive programs for pollution abatement in cooperation with the control agencies of each state. An intensive study now is being made of the streams in the principal drainage basins of the country to show the existing pollution loads and determine essential uses of the waters for various purposes. Water quality criteria will

* Abstract of this paper will appear in the April issue of the Journal.

then be established and degrees of treatment assigned for each source of waste discharge.

In assembling the data upon which recommendations for corrective measures will be based, the Service is cooperating closely with the control agency in each state and with industry through the National Technical Task Committee on Industrial Waste. This Committee has been organized to assist as a coordinating and advisory body on technical matters involved in the treatment of industrial wastes. Four "Task Groups"** have been established to function as working committees in carrying out this program. Task Group III consists of chemical processing industries, with subcommittees representing the tanning,** pulp and paper, textile, rubber, chemical, and electroplating industries. The immediate objective of this joint government-industry approach to the problem will be:

1. To compile a source list of procedures that can be utilized in the treatment and control of industrial wastes.
2. To assemble a tabulation of research projects currently in progress or contemplated, which deal with the utilization of waste products or their treatment and disposal.
3. To make available a listing of problems of treatment and disposal for which a solution is not now available, and which are common to a number of industries.

The primary purpose in the development of these comprehensive programs is to provide an accurate picture of the extent to which pollution has damaged the country's water resources, and to outline in detail the measures that must be adopted to restore and maintain them in a satisfactory condition. Specific requirements for the treatment of industrial wastes will be made a part of these programs, which will be administered by the local control agencies.

It is now generally accepted that surface waters must be utilized to a reasonable extent as a medium for disposition of liquid wastes. Under this premise, consideration will be given to many factors in determining abatement measures for specific wastes. Undoubtedly, location of a particular plant and essential usage of the receiving body of water will largely influence the type and degree of treatment necessary.

As these programs become operative, all producers of industrial waste will be confronted with a disposal problem. Plants located where ample dilution is available can anticipate that only a minimum of treatment may be required; others, located in built-up areas or adjacent to small streams will be compelled to provide more efficient facilities.

Compliance with these requirements will be costly and a matter of serious concern to such highly competitive industries as tanning. Present day know-

**Dr. Fred O'Flaherty, Director of the Tanners' Council Research Laboratory at the University of Cincinnati, is the representative of the Tanning Industry on Task Group III.

ledge of tannery effluents offers little encouragement in the possibility that saleable by-products may be recovered to at least partially defray the cost of treatment. Continued technological improvement in the manufacture of leather may disclose means whereby certain wastes can be substantially reduced or entirely eliminated by conversion to re-usable products.

As an initial step toward the solution of a specific problem in waste disposal, it is important that complete information be available concerning the quantity and character of all wastes produced in the plant. A thorough survey undoubtedly will disclose where a substantial reduction in total volume can be obtained through conservative use of water without interfering in any way with the quality of the product. The survey also may demonstrate the practicability of recirculation of certain rinse waters, or segregation for direct discharge to the stream. The cost of treatment will be roughly proportionate to the volume to be handled and consequently it is of the utmost importance that the quantity of raw wastes be reduced to an absolute minimum.

The method of disposal is a matter for decision by each plant. In some instances, it may be possible to arrange for disposition of the wastes through the public sewer system. Most municipalities, however, will not accept wastes that may clog or otherwise detrimentally affect the sewers, or which will interfere with normal functioning of the sewage treatment plant. Frequently these objections can be overcome by installation of pre-treatment measures for removal of the undesirable characteristics prior to discharge to the sewer.

At tanneries where disposal is made directly to a body of water, the wastes must be conditioned in accordance with standards to be established for that locality. These standards undoubtedly will necessitate substantially complete removal of the settleable solids as a minimum requirement. A higher degree of treatment for reduction of color and excessive oxygen demand may be required for plants located in restricted areas. A number of factors will influence the type of facilities that may be employed to accomplish the necessary treatment. The kind of leather manufactured, volume of waste and land available for disposal units vary with every tannery, and will largely determine the procedure that can be utilized to the best advantage. Each problem must be studied intelligently in order that an efficient and economical installation can be provided.

The programs now being developed envisage the restoration of surface waters to a reasonable standard of cleanliness. Industry is being asked to do its share toward achievement of this objective by providing satisfactory disposal of its process wastes. Every effort will be made by the control agencies to secure the requisite abatement by cooperative means. The failure or refusal of industry to recognize its responsibilities can and will lead to the application of drastic penalties.

Canaigre Investigations

VIII. Preparation of Tanning Extracts by Continuous Countercurrent Extraction*†

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Previous papers from this Laboratory have reported the progress made in developing a domestic source of tanning extract from roots of canaigre (*Rumex hymenosepalus* Torr.). This paper, reporting further developments, describes several possible processes for the commercial production of tanning extracts from canaigre, in all of which continuous countercurrent extraction is used for separating the tannin from the roots. The economics of these various processes have not been thoroughly investigated as yet, and the qualities of the extracts produced have not been completely evaluated. Laboratory tests have shown, however, that at least one of these processes produces tanning extracts which could replace several vegetable tanning extracts in commercial use in this country.

The background of the canaigre project has been covered thoroughly in articles by Frey and Sievers [1] and Rogers and Russell [2]. The major difficulties in the extraction of canaigre are caused by the starch it contains. If the material is heated to a temperature at which the starch gelatinizes with water, the tannin becomes much more difficult to extract and, in addition, the liquors are much more difficult to clarify. Any process to extract tannin from canaigre must take these factors into consideration.

There are at least two strains of the canaigre plant, one having a root with a yellow interior and the other a root with a reddish interior. Since the latter is much higher in tannin content and purity, and since this strain is more easily extracted and gives liquors which are more easily clarified, reddish roots with a tannin content of about 37 per cent and a purity of about 67 were used in practically all these experiments.

EXTRACTION OF TANNIN FROM CANAIGRE

Before the production of tanning extract from canaigre as an integrated process is discussed, it might be well to describe the extraction step, because the solvents and conditions used in the extraction influence the later steps of the process.

Previously an attempt had been made to leach canaigre in the conventional way used for tannin-bearing barks and woods, that is, percolating hot water

*Report of a study made under the Research and Marketing Act of 1946.

†Presented at the American Leather Chemists Association Meeting, French Lick, Indiana, May 31–June 2, 1950.

**One of the laboratories of the Bureau of Agriculture and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

through a stationary bed of shredded canaigre. It soon became apparent that the hot water gelatinized the starch, making the bed impermeable and the tannin difficult to extract. Cooler water improved the permeability of the bed slightly, but reduced the extraction efficiency materially because the shreds had to be large to permit the water to percolate through the bed. Beebe, Cordon and Rogers [3, 4] published the results of laboratory and small-scale work on a centrifugal filtration method of extraction with water. Although this worked well in the laboratory, when this method was extended to pilot-plant scale, the rate of filtration through a perforate bowl was rather slow. Centrifugal separation with a solid bowl was not thoroughly investigated because other means of processing appeared to be less costly. Accordingly, a vibrating screen and continuous rotary press of the conical type were substituted for the centrifugal filter. This method did not give quite as high extraction efficiencies as that previously mentioned mainly because considerable tannin was lost in fines, which were carried into the head liquor. The equipment and labor costs of handling both liquids and solids in this manner in a countercurrent batch operation on a large scale would be unduly great.

Luvisi and Rogers[5] and Luvisi, Cordon, Beebe and Rogers [6] have published results of laboratory-scale extraction of canaigre with organic solvent-water solutions, particularly acetone-water solutions. The present authors, in extending these studies to pilot-scale operations, have used isopropyl alcohol instead of acetone in most cases because it can be used in more dilute solution and because it is more feasible for commercial operations. The stationary vat leaching of large shreds of canaigre with dilute organic solvents instead of water was tried by Rieder et al [7]. The solvent improved the permeability of the bed, and uniform shreds relatively free of fines made it even more satisfactory. But even though the recovery of tannin was increased somewhat over that obtained by water extraction, it still was not enough to make this process commercially feasible. The continuous countercurrent extraction methods presented in this article are the result of attempts to make the process less expensive.

Before the study was started, a survey was made of the equipment used in the solvent extraction field. The common types are (1) the basket or paternoster type and a horizontal development of this called the Rotocel, (2) the vertical-column tray type, (3) screw or Redler conveyors, and (4) the paddle-wheel type or Kennedy extractor. The only one definitely unsuitable was the basket type, since it depends on percolation of the solvent through a stationary bed of fine material in a short time. As we had proved previously, this can not be done with canaigre because it swells and becomes impervious to the solvent. Any of the other types of extractors might be satisfactory for the purpose, but since a Kennedy extractor was available at the Laboratory it was used for this work.

The Kennedy extractor (Figures 1 and 2) consists of a long narrow horizontal chamber having a jacketed bottom made up of a series of semi-

cylindrical cells. In each cell is a paddle wheel or rotor with four perforated blades turning slowly in such a way that the solids are propelled along the extractor. As modified from the original design, the extractor used now has perforated weirs between the cells and a scraper blade on each paddle wheel to scrape the solids off the paddle wheel into the next cell. The solids are metered into one end of the extractor by a grooved disk feeder, and removed at the other end by a drag conveyor. The solvent is fed by a positive displacement metering pump through a rotameter into the last cell before the drag conveyor, and by a slight slope of the extractor is caused to flow in a direction countercurrent to that of the solids. It is discharged as head liquor at the opposite end of the extractor. The extractor has 14 extraction cells and 1 filter cell, but it is adaptable so that fewer can be used if desired.

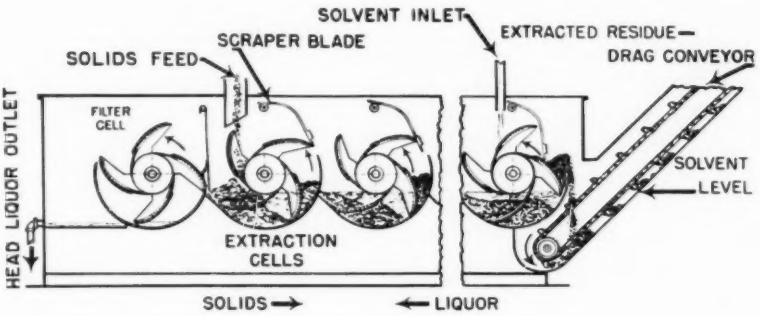


Figure I. Sectional View of Kennedy Continuous Countercurrent Extractor

A series of experimental runs was made in which the solvents were (1) distilled water, (2) an aqueous solution containing 18 per cent by volume of isopropyl alcohol and (3) another aqueous solution containing 25 per cent by volume of acetone. Other variables studied were the method of cutting and the size of the solids, retention time, liquid-to-solids ratio, number of cells necessary for the extraction and temperature of the solvent. In all cases, moisture analyses were made on the solids as fed and on the spent marc, and tannin analyses were made on the solid feed, the spent marc and the head liquor. The samples of fresh and spent materials were extracted for analysis by an improved procedure,[5] and all the solutions and liquors were analyzed for tannin by the official methods of the A.L.C.A.[8]. The details of these 52 runs are the subject of a paper devoted solely to extraction[9], but we shall summarize the conclusions.

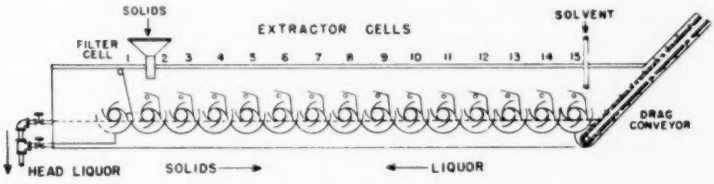


FIGURE 2. DIAGRAM OF KENNEDY CONTINUOUS COUNTERCURRENT EXTRACTOR

1. *Preparation of roots*: For extraction with aqueous organic solvents, the dried roots were ground in a rotary knife cutter (Ball and Jewell type) with a 1/8 inch screen, the ground material was passed over a 20-mesh screen, and the material held on the screen was reground. This preparation gives good extraction and a minimum amount of fines in the head liquor. For water extraction, the dried roots were ground in the rotary knife cutter with a 1/16 inch screen. Although this preparation causes somewhat more fines to be carried into the head liquor, the finer comminution is necessary for water extraction. Other methods of grinding might produce even a better preparation for extraction.

2. *Retention time of the solids in the extractor*: Retention times of 60 to 150 minutes were investigated. It was concluded that for extraction with aqueous organic solvents, a retention time of 90 minutes was most practical, but for extraction with water a retention time of 120 minutes was better.

3. *Liquid-to-solids ratio*: With dilute organic solvents a liquid-to-solids ratio of 10 to 1 gave about as good an extraction as could be attained. With water, slightly higher ratios might be more economical.

4. *Number of cells required*: For extraction with dilute organic solvents, only 10 Kennedy cells were useful. With water as the solvent, extraction takes place in all 14 cells.

5. *Effect of different solvents and of temperature*: With 18 per cent by volume aqueous isopropyl alcohol at 112° F. and 25 per cent by volume aqueous acetone at room temperature, 94 per cent of the tannin was recovered in the head liquor of the Kennedy extractor under the optimum conditions described. Although 25 per cent acetone gave tannin recoveries higher by about 1 to 2 per cent than those given by the 18 per cent alcohol, it is believed that the use of this solvent on a commercial scale would be undesirable because of the difficulties and expense involved in handling this more inflammable material. Increase in temperature of the acetone had little if any beneficial effect. The effect of increasing the temperature of the isopropyl alcohol above 112°

F. has not been thoroughly investigated. Below 112°F., however, the extractive powers of this solvent decrease appreciably.

With water at 112°F. as the extractant, 73 per cent of the tannin was recovered. At elevated temperatures, recoveries up to 78 per cent have been reached. These values cannot be compared with those of Beebe et. at. [3, 4] because the present figures are based on the improved analyses of the roots by the Luvisi method[5]. If the earlier investigations had been based on this improved analytical procedure, the recoveries of tannin they reported would have been somewhat lower.

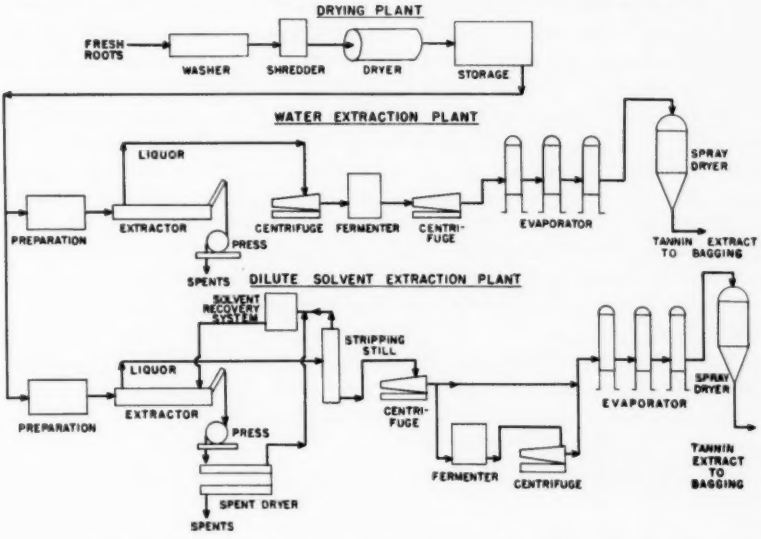
Because these solvents give greater recovery of tannin it might appear that extraction with organic solvents would be the best process to use commercially. But recovery is not the complete picture. The quality of the tanning extract produced and many economic factors must be considered. The tanning quality of extracts prepared with different solvents and at different temperatures is discussed in a paper presented by Beebe et al. at the meeting of the American Leather Chemists Association, French Lick, Ind., May 31-June 2, 1950. The economics cannot be worked out accurately because of uncertainty regarding the price of the raw material in terms of tannin content and because the possibilities of utilizing the spent canaigre and other byproducts have not yet been determined. It must be remembered that if organic solvents are used, the effective solvent-recovery system required will cause additional expense. Before a definite commercial choice can be made, the economic aspects of the various processes must be thoroughly evaluated.

Production Process—Fresh Roots to Powdered Extract

We shall now discuss the over-all process from fresh roots to powdered tanning extract. It must be kept in mind that the choice between water and aqueous organic solvent will determine the character of the rest of the process. The flow sheets for the two processes are shown in Figure 3. The danger of iron contamination of tannin must be remembered; all piping and metal equipment that come in contact with the canaigre roots or the liquors should be copper, brass, or stainless steel.

Since it appears to be most economical to operate the extraction plant on a 12-month basis, and the canaigre harvesting season lasts only 3 months, the processes described here include shredding the roots and drying them for storage. Laboratory tests have shown that to keep the shredded roots from molding and fermenting they must be dried to a moisture content below 16 per cent [10]. Freshly harvested roots can be washed in any type of commercial potato or sugar beet washer. The choice of a shredder depends on the drying method. Preliminary drying investigations have indicated that canaigre can be satisfactorily dried in either of two ways. Cut into shreds

FIGURE 3
FLOW SHEETS FOR RECOVERY OF TANNING EXTRACTS FROM CANAIGRE ROOTS



they can be dried in a belt dryer in which aîr at 150° F. is blown down through a thin bed of shreds. Or, ground in a rotary knife cutter or sharp knife hammer mill equipped with a ¼-inch or ½-inch screen, they can be dried in a parallel current rotary kiln dryer with air at 500 to 600° F. inlet temperature. The latter drying method, of course, is less expensive; for Kennedy extraction wotk it produces a very satisfactory product.

When water is used as the extractant, the spent material from the extractor is passed through a press and the press liquor is returned to the extractor with the fresh solvent. The pressed spent roots do not have to be dried. Because of their high starch content, they might find various uses.

The water extract is passed through a centrifuge, which decreases the insoluble material to 2 or 3 per cent of the total solids in the clarified liquor. After centrifuging, the water-extracted liquor, which has a purity of about 50, is sterilized by heating to 200° F., cooled to 87° F. and fermented to remove the sugars, thereby increasing the purity. This fermentation was developed by Cordon, Beebe and Rogers [11]. They used an *Aerobacter aerogenes* (designated as WH), which ferments the sugars in tannin liquors in about 16 hours, producing mainly 2,3-butanediol, and smaller quantities of acetoin, ethanol, and organic acids [12]. By this operation, the purities of water-extracted liquors can be increased to 60 or 70. After fermentation, the liquors are recentrifuged to remove insolubles produced during this stèp.

The clarified liquors are transferred to a standard multiple-effect evaporator, and concentrated either to a liquid extract of about 60 per cent solids or to a liquid having a solids content suitable for spray drying. Spray drying has been successfully accomplished with canaigre extracts ranging from 25 to 50 per cent in solids content and with inlet air temperatures of 400 to 600°F.

When dilute organic solvents are used, the following modifications must be made in the process just described for water extraction. The pressed spent roots must be dried to recover the valuable solvent. Before the head liquor from the extractor is centrifuged, it must be passed through a stripping still to recover the solvent. It has been calculated and demonstrated that if 18 per cent by volume of aqueous isopropyl alcohol is used as the extractant, when two-fifths of the solution has been vaporized, the distillate will contain all the alcohol at a concentration of about 40 per cent. This distillate can be diluted and recycled to the extraction system.

Since solvent-extracted liquors have a purity of 59 to 63, there is a question as to whether it is necessary to increase the purity by fermentation. Luvisi et al.[6] state that fermentation is not necessary. If fermentation is not used, the second centrifuging is eliminated.

The remainder of the process is the same as that for water-extracted liquors.

Production of Extracts for Tanning Tests

To produce a sufficient quantity of canaigre extract for tanning tests, several production runs were made on the Kennedy extractor with different solvents and conditions. The four solvents used were water, 10 per cent by volume aqueous isopropyl alcohol, 18 per cent by volume aqueous isopropyl alcohol, and 25 per cent by volume aqueous acetone. Four water runs were made, one each at temperatures of 114°, 150° and 170° F., and one in which the water was used at 175° at the solids discharge end of the extractor and cooled until it was 115° F. at the head liquor end. Table I gives the conditions, tannin recoveries, and analyses of the powdered extracts produced. The duration of the runs varied somewhat but averaged 24 hours. The roots used were a reddish strain having 37 per cent tannin content and a purity of 67; they had been dried in a belt dryer with air at 150° F. The dried roots were fed into the extractor at a rate of approximately 2.5 pounds per hour. The head liquors, which were not collected until the extractor had reached equilibrium, were carried through all the stages of the processes described above, including fermentation in every case except the run in which 25 per cent acetone was used.

Table I shows that the actual recovery of tannin in the head liquors for the water runs ranged from 62 to 71 per cent. For the 18 per cent isopropyl alcohol run, the recovery was 88 per cent, and for the 25 per cent acetone run, 86 per cent. Unfortunately, extraction data could not be obtained for the 10

TABLE I

Spray-Dried Canaigre Extracts Made for Tanning Tests

Conditions of Extraction					Head Liquor Produced		Spent Roots	Over-All Balances*		Analyses of Powdered Tanning Extracts					
Run No.	Solvent	Temp.°F.	Retention Time Min.	Liquid to Solids Ratio	Tannin Recovery %	Purity	Tannin Content %	Tannin %	Soluble Solids %	Total Solids %	Soluble Solids %	Insoluble Solids %	Non-tannin %	Tannin %	Purity**
CA31W47	Water	114	120	10.6	67	58.2	17.2	90	95	96.4	95.1	1.3	35.1	60.0	63.0
CA31W48	Water	150	90	10.8	71	57.5	15.5	89	93	96.0	92.3	3.7	28.9	63.4	68.7
CA31W45	Water	170	90	10.2	66	58.5	13.2	82	87	95.1	92.3	2.9	34.7	57.5	62.4
CA31W46	Water	175 on spent roots to 115 on feed	90	13.1	62	57.4	15.4	82	86	96.1	94.0	2.1	33.2	60.8	64.7
CA31E45	10% Isopropyl alcohol	112	90	——	—	59.4	7.4	—	—	96.4	95.0	1.5	32.3	62.7	66.0
CA31E44	18% Isopropyl alcohol	112	90	11.1	88	62.5	5.8	95	100	96.9	93.9	3.0	28.7	65.3	69.5
CA31C3	25% Acetone	80	90	11.9	86	62.2	4.8	92	98	94.9	93.6	1.3	35.2	58.5	62.4**

*Over-all balances are calculated as the amount of the constituent recovered in the head liquor plus the amount in the spent roots divided by the quantity originally in the roots.

This value is expressed as percentage.

**The increase in purity shown in this column is the result of fermentation of the head liquors. All the liquors were fermented except that from Run C3.

per cent isopropyl run. It will be noticed that the tannin recoveries are not quite so high as those reported earlier in this paper as being attainable and also that the effect of increasing the temperature of the water on the extraction is not clearly shown by these results. The reasons for this are twofold. First, a certain deviation is allowable in any one run because of errors in experimental measurements and in analyses which are unavoidable in working with natural products. Second, the data reported are for a series of individual runs made solely to produce tannin for tanning tests. Because of the comparatively large quantities of tannin required, the extractor was operated overnight unattended which precluded the rigid controls used in our experimental work. It will be seen that the recovery figures in Table I which may be considered low are accompanied by low tannin balances and low soluble-solids balances, indicating inaccuracies in measuring or losses of material. The figures reported as conclusions in the earlier part of this paper are the results of a series of many experimental runs. The effect of increasing the temperature of water for extraction is indicated better by the column in Table I giving the tannin contents of the spent roots. These data show clearly that the higher the temperature of the water the less the amount of tannin in the spent roots.

Summary

A process has been presented for producing tanning extract from canaigre roots by continuous countercurrent extraction with various solvents. The process includes shredding and drying the fresh roots, preparing the dried material, extracting with water or with dilute aqueous solutions of organic solvents, stripping to recover solvent when necessary, centrifuging, fermenting if necessary, clarifying, concentrating and spray drying to powdered tanning extract. Details are reported of runs made to prepare sufficient tanning extract by each of these processes to permit evaluation of the quality of the tannin by tanning tests. Economic evaluation of these processes cannot yet be made because of the uncertainty of the price of the raw material in terms of tanning and because uses have not yet been found for the byproducts.

Acknowledgment

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Canaigre Investigations

IX. Laboratory Tannage of Heavy Leather*†

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Abstract

Blocks of steer hide were tanned in the laboratory with canaigre extracts made by several methods. All the leathers produced were well tanned, firm, well filled, and of good appearance. Extracts made with water gave better penetration into the hide, and formed less sludge during tannage than those made with solutions of water and isopropyl alcohol or acetone. An extract made with water at 150° F. gave better tanning results than those made at either higher or lower temperatures.

Introduction

Final evaluation of a new tanning material requires a large-scale commercial tanning test. A sufficient amount of canaigre extract for such a test has not yet been made. However, sufficient extract was on hand to tan, on a laboratory-scale, several hides cut into blocks of suitable size. Although such a test is not equivalent to a large-scale test, it indicates the type and quality of leather which can be made and gives a comparison of the tanning properties of different types of extract.

*This is the ninth of a series of papers reporting various phases of cooperative investigations of canaigre as a source of tannin by the Bureau of Agricultural and Industrial Chemistry and the Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration, United States Department of Agriculture.

**One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Administration, United States Department of Agriculture.

†Report of a study made under the Research and Marketing Act of 1946.

Experimental Work

Two series of tanning tests were made. In the first series, two kinds of canaigre extracts were used; one was a liquid extract made by water extraction, and the other a powdered extract made by extraction with an 18 per cent solution of isopropyl alcohol in water.* One tannage was made with each of these extracts. As there was an insufficient amount of the alcohol extract, a slight amount of similarly prepared extract with a somewhat higher purity was used during the last week of tannage. A third tannage was made with a blend of 40 per cent of the alcohol extract, 30 per cent of sulfited quebracho, and 30 per cent chestnut wood extract. A fourth tannage was a control tannage made with a blend of equal parts of quebracho and chestnut extracts. Each of the vats used held one side of leather cut into 15 by 12 inch blocks. Four hides, previously limed, unhaired, and delimed slightly, were cut into blocks of this size. Each hide served for two comparative tannages. Alternate blocks from each side were put into two vats so that each vat contained a mixture of right and left-hand pieces and each piece was matched by a piece in the other vat from a corresponding position on the other side of the animal. The weight of tanning liquor was about three times the weight of the wet white hide pieces.

Tannage was started in a liquor containing 0.5 per cent tannin. Every day one-third of the liquor was discarded, and enough settled, clear stock liquor added to raise the strength 2° barkometer over that of the preceding day. Each day for the first 10 days, the pH was adjusted to 4.5 by addition of lactic acid. During the first few days, there was a rapid raise in pH, caused by the lime in the hides and the lack of buffering material in the liquors, so that the actual pH of tanning ranged from about 5.5 in the tail liquor to 4.5 in the liquor after 10 days. For the remainder of the time, the pH was determined but unadjusted. Every few days, until the pieces were completely "struck through" by the tannin, cuts were made in pieces of comparative thickness and location to determine the rate of penetration of the tannin. The time of tannage was 6 weeks. The strength of the final liquors was 65° barkometer.

Four more tannages were included in the first series. The liquors for these tannages were the discarded liquors from the first tannages except for the fresh liquors used in the final week. They were started 1 week after the first four tannages. The amount and composition [1] of all fresh and discarded liquors from all tannages were determined and recorded.

This method of tannage is different from the countercurrent system used commercially, which could not be used in these tests because of shortage of materials. Although the laboratory system might have a slight advantage over the other because impurities are not accumulated, it is, on the whole,

*Canaigre extracts prepared by extraction with water are subsequently referred to as water extracts; those prepared by extraction with a solution of isopropyl alcohol in water are referred to as alcohol extracts; the term solvent extract refers to an extract prepared by extraction with a solution of either isopropyl alcohol or acetone.

disadvantageous. In practice, the liquors are mellowed by contact with partly tanned leather and consequently are less astringent when applied to fresh hide. In addition, they accumulate buffer salts, so that it is possible to adjust the pH without sharp peaks and dips.

There was a fairly heavy growth of mold on the liquors. Although this was partly due to the method of tannage, apparently canaigre has more of a tendency to mold than do other tanning materials. There was less mold on the controls than on the canaigre tannages. A marked difference was noted in the sludge or deposit formed in the liquors. The amounts formed in the liquors prepared from water-extracted canaigre, the blend, or the control liquors were normal for usual tanning materials. In the solvent-extracted canaigre liquors, however, a heavy sludge formed. In these liquors, the apparent sappage each day was considerably more than in the other liquors, because in addition to what was taken up by the leathers, solids were lost by settling. Therefore, to maintain the strength of the solvent liquor, much more stock liquor was required than for the other tannages.

After tannage, the leathers were washed. A marked difference was noted between the pieces tanned with solvent extract and those of the other tannages. All the pieces were easily washed except the pieces tanned with the solvent extract, and they required vigorous scrubbing with a brush. The pieces were oiled lightly, dried, and weighed. They were then bleached, oiled and dried by the usual procedure at a commercial tannery, along with a pack of regular leather being given these treatments. They were pressed at the laboratory and conditioned at 73° F. and 50 per cent relative humidity, weighed and measured for thickness. Tensile strength was determined on a Tinius Olsen "Plastiversal" testing machine of 0-500 pounds capacity. Elongation was measured on the same machine at a load of 100 kilograms per centimeter width. Water absorption was measured by the method of Whitmore and Downing[2]. Shrinkage temperatures were determined by the Theis Shrinkage Tester, according to the specifications of the provisional method of the American Leather Chemists Association[1] (1942); no adjustment of the pH was necessary. Determinations of pH and chemical analyses were made by the official methods of this Association.

In the second series of tanning tests, eight types of canaigre extracts, prepared by Rieder et al.[3] were used. Five of these were prepared by water extraction—one at 112° F., one at 114°, one at 150°, one at 170°, and one at temperatures ranging from 175° on the tail to 115° on the head leach. Three extracts were prepared by extraction with solutions of organic solvents: one with an 18 per cent solution of isopropyl alcohol at 112° F.; one with a 10 per cent solution of isopropyl alcohol at 112° F.; and one with a 25 per cent solution of acetone at 80°. Another tanning test was made with an extract prepared by treating the 18 per cent alcohol extract with 3 per cent sodium sulfite on a total solids basis, to prevent formation of insoluble material.

Since smaller amounts of material were available for the second series of tests than for the first series, they were made in battery jars each holding three 10.5 by 8 inch-pieces. A hide similar to those used in the first series of tests was cut into blocks of this size, and two blocks from the bend portion and one from the belly or shoulder portion were tanned in each jar. The distribution of pieces was such as to give fairly comparable results. The liquor-to-hide ratio was about the same as in the first series. The tanning method was, in general, the same as in the first series except that instead of increasing the barkometer strength of the liquors to a definite extent each day, stock liquors were added in amounts that gave the same addition of tannin to each vat. In these tests, mold growth was prevented by addition of a small amount of preservative (phenyl mercuric acetate).

As in the first series, a tendency for sludge formation was noted in the liquors from the solvent extracts and, to a smaller extent, in the liquors from the water extracts made at 170° F. and 175° F. Because of the small size of the vats used in this test, the comparatively vigorous rocking motion, and the mixing motion caused by removing the hide pieces, the liquors became well mixed before the sapped portion was taken off. Therefore, a large proportion of the sediment formed each day was removed and did not accumulate as it did in the first test, and as it would in actual tanning.

The leathers were weighed wet, after removal of superficial water with a towel. Since an opportunity became available to have these leathers finished in a tannery, in conformity with usual tannery practice, without an intermediate drying after tannage, they were handled in this manner. In this respect, they differed from the leathers in the first series, which were dried between tanning and finishing.

TABLE I

Analyses of Extracts*—First Series of Tanning Tests

	Total Solids %	Soluble Solids %	Insolubles %	Non tannin %	Tannin %	Purity Tannin Soluble Solids
Alcohol extract I	95.0	91.1	3.9	36.0	55.1	60.5
Alcohol extract II	96.1	87.6	8.5	29.6	58.0	66.2
Water extract	46.1	45.6	0.5	18.2	27.3	60.0
Blend	45.0	43.2	1.8	13.2	30.0	69.4
Control	50.7	48.3	2.4	11.7	36.6	75.7

*Alcohol extract I was prepared by extraction with 18% isopropyl alcohol solution at 112-116° F.

Alcohol extract II was a composite of three extracts prepared by extracting (1) with 18% isopropyl alcohol-water, (2) with 15½% isopropyl alcohol-water, and (3) with 25% acetone-water mixtures.

Liquors from which alcohol extract II and the water extract were made were fermented to improve purity, but those used in preparing alcohol extract I were not fermented. Only slight amounts of extract II were used for strengthening liquors during the last week of tannage.

The blend consisted of 40% of the alcohol extract, 30% chestnut wood extract, and 30% quebracho extract.

The control was a blend of 50% quebracho extract and 50% chestnut wood extract.

Data and Calculations

Table I shows the analyses of the extracts used in the first series of tanning tests. There is little significant difference in the analyses of the three canaigre extracts.

The time required for complete penetration of tannin through pieces of hide of equal thickness (¼") was: in the first round, water-extract, 17 days; alcohol-extract, 20 days; blend of canaigre, chestnut, and quebracho, 19 days; and control, chestnut and quebracho, 16 days. In the second round, it was: water-canaigre, 20 days; alcohol-canaigre, 23 days; blend, 20 days; and control, 19 days.

Table II shows some data on yields and tannin consumption for the first series of tests. The yield figures are somewhat higher for the first round of the alcohol extract than for the other extracts. The tannin consumption reported is tannin either taken up by the leather or lost. It is calculated by determining the total tannin in the stock liquor used and subtracting from this figure the tannin discarded and that remaining in the liquors at the end of tannage. This figure divided by the grams of leather gives the tannin consumption per gram of leather tanned. If calculated on the basis of the rough dry leather, the tannin consumption is due to tannage alone; if calculated on the basis of the finished leather, it is due to tannage plus oils and loading materials. The tannin consumption for the first round of the alcohol-extract tannage was considerably higher than that for the other tannages. In the second round, there were only slight differences in the results obtained.

TABLE II

Leather Yields and Tannin Consumption—First Series of Tanning Tests

	Leather Yields		Tanning Consumption per gram of leather	
	White Weight to Rough Dry Leather %	White Weight to Finished Leather %	Rough Dry Leather grams	Finished Leather grams
Water extract				
1st round	60.3	69.8	0.42	0.36
2d round	60.1	69.7	0.42	0.34
Alcohol extract				
1st round	63.2	73.2	0.57	0.50
2d round	61.5	69.3	0.42	0.34
Blend				
1st round	57.9	70.4	0.49	0.41
2d round	57.7	69.3	0.49	0.36
Control				
1st round	58.8	69.9	0.46	0.39
2d round	58.6	69.7	0.46	0.38

Some chemical and physical data on the leathers in the first series of tests are shown in Table III. Although the yield of the alcohol-extract leather (Table II) was higher than that of the water-extract leather, the combined tannin in the former leather was only 26.5 per cent as compared with 30.4 per cent in the latter leather. The higher yield was due to the larger amount of soluble matter—30.8 per cent—as compared with 23.7 per cent for the water-extract leather. The remaining data in the chemical analyses require no comment. They are normal for this type of leather. There are some variations in the data for tensile strength and water absorption. However, these methods are not sufficiently accurate for these variations to have appreciable significance.

TABLE III

Chemical and Physical Data on Leather—First Series of Tanning Tests

	Water Extract		Alcohol Extract		Blend		Control	
	1st Round	2d Round	1st Round	2d Round	1st Round	2d Round	1st Round	2d Round
Oils and fats, %	4.2	6.4	5.7	7.0	6.1	8.0	5.1	5.8
Insoluble ash, %	0.3	0.3	0.4	0.3	0.4	0.4	0.4	0.4
Soluble matter, %	23.7	23.2	30.8	22.7	23.5	25.3	22.2	27.6
Hide substance, %	41.4	41.0	36.6	41.1	40.0	40.1	40.5	39.5
Combined tannin, %	30.4	29.1	26.5	28.9	30.0	26.2	31.8	26.7
Degree of tannage, %	73.4	70.9	72.4	70.3	75.0	65.3	78.5	67.6
Soluble nontannin, %	15.7	16.5	18.4	16.6	17.0	17.2	15.1	17.3
Soluble tannin, %	8.0	6.7	12.4	6.1	6.5	8.1	7.1	10.3
Total ash, %	5.6	5.4	5.7	5.4	6.3	5.9	5.8	5.5
Epsom salts, %	7.1	6.5	7.1	7.0	7.7	7.4	6.6	6.5
Glucose, %	3.8	3.8	4.8	4.4	5.0	4.6	5.0	5.1
pH,	3.1	3.8	3.0	3.1	3.3	3.9	3.9	3.4
Shrinkage temperature, C°	82	89	84	87	86	86	86	85
Tensile strength, lbs./sq. in.	4500	4440	4610	5550	3890	4960	5070	——
Elongation, %	19	20	18	18	21	18	16	—
Water absorption								
30 minutes (A), %	24	25	35	27	37	40	48	—
24 hours (B), %	44	50	51	50	54	56	60	—
Ratio A/bx 100	55	50	70	54	69	72	80	

Table IV gives the analyses of the extracts used in the second series of tanning tests. The most significant data are the figures on purity. The comparatively high purities of some of the extracts were due to a fermentation process, which reduced the amount of nontannins.

The time taken for complete penetration of tannin through pieces of hide of equal thickness in the second test was: for the water extracts prepared at 112° F., 23 days; at 114°, 22 days; at 150°, 22 days; at 170°, 23 days; and at 115–175°, 24 days. For the solvent extracts, the time was: 18 per cent alcohol,

28 days; 10 per cent alcohol, 24 days; acetone, 26 days, and sulfited liquor, 16 days.

TABLE IV

Analyses of Extracts—Second Series of Tanning Tests

	Total Solids %	Soluble Solids %	Insolubles %	Non-tannin %	Tannin %	Purity* Soluble Tannin Solids
Water, 112° F.	95.8	94.9	0.9	40.4	54.5	57.4
Water, 114° F.	96.4	95.1	1.3	35.1	60.0	63.1
Water, 150° F.	96.0	92.3	3.7	28.9	63.4	68.7
Water, 170° F.	95.1	92.2	2.9	34.7	57.5	62.4
Water, 115-175° F.	96.1	94.0	2.1	33.2	60.8	64.6
18% isopropyl alcohol 112° F.	96.9	93.9	3.0	28.7	65.3	69.5
10% isopropyl alcohol, 112° F.	96.4	95.0	1.4	32.3	62.7	66.0
25% acetone, 80° F.	94.9	93.6	1.3	35.2	58.4	62.4

*The higher purities of extracts 2-7 are due to the fact that their sugars were removed by fermentation, thus lowering the nontannins.

Table V gives the yields and tannin consumption of the leathers in the second series of tests. The rough weights of the leather are the wet weights instead of the dry weights as in the first series. Only slight differences in tannin consumption are shown. However, the system used in the two tannages was different. In the first series, the strength of the liquors was maintained at a definite figure, irrespective of losses, but in the second series, a definite amount of tannin was added to each vat without regard to the final strength. In the second series, differences in the tannin consumption were

TABLE V

Leather Yields and Tannin Consumption—Second Series of Tanning Tests

	Leather Yields		Tannin Consumption per gram of Leather	
	White Weight to Wet Rough Leather %	White Weight to finished Leather %	Rough Wet Leather grams	Finished Leather grams
Water, 112° F.	114.0	75.4	0.13	0.39
Water, 114° F.	115.8	76.3	0.12	0.36
Water, 150° F.	120.2	89.2	0.14	0.38
Water, 170° F.	113.7	74.5	0.12	0.37
Water, 115°-175° F.	115.3	77.6	0.12	0.34
18% isopropyl alcohol, 112° F.	112.7	74.4	0.15	0.40
10% isopropyl alchol, 112° F.	113.2	82.9	0.13	0.36
25% acetone, 80° F.	112.9	73.6	0.12	0.37
Sulfited liquor	114.8	72.6	0.12	0.37

TABLE VI

Chemical and Physical Data on Leathers—Second Series of Tanning Tests

	Extract Prepared by Water Extraction at					Extract Prepared by Water-Solvent Extraction			
	112°F.	114°F.	150°F.	170°F.	115–175°F.	18% Iso-propyl Alcohol	10% Iso-propyl Alcohol	25% Acetone	Sulfited
Oils and fats, %	5.2	5.0	5.3	5.8	5.3	4.3	4.1	4.1	6.5
Insoluble ash, %	0.3	0.2	0.2	0.2	0.2	0.2	0.3	0.2	0.2
Soluble matter, %	30.1	32.7	31.9	30.4	32.7	28.4	30.8	27.8	30.5
Hide substance, %	37.5	35.4	33.3	38.1	34.4	38.1	36.5	39.6	38.0
Combined tannin, %	26.9	26.7	29.3	25.5	27.4	29.0	28.3	28.3	24.8
Degree of tannage, %	71.8	75.2	87.7	66.9	79.7	76.1	77.5	71.5	65.3
Soluble nontannin, %	20.8	21.7	21.5	20.0	22.5	18.1	20.3	18.1	20.5
Soluble tannin, %	9.3	11.0	10.4	10.4	10.2	10.3	10.5	9.7	10.0
Total ash, %	5.8	6.5	6.5	6.1	6.9	5.3	6.1	4.8	5.7
Epsom salts, %	7.4	7.6	7.7	7.5	8.2	7.1	7.3	6.5	7.1
Glucose, %	7.6	7.8	7.4	7.1	8.5	6.4	7.2	6.3	7.8
pH	3.6	3.6	3.5	3.6	3.6	3.6	3.6	3.8	3.7
Shrinkage temperature, C°	84	86	84	85	85	86	82	85	83
Tensile strength, lbs. / sq. in.	3500	3300	3200	3500	2600	3000	2700	3100	3200
Elongation, %	17	21	14	17	23	14	23	17	20
Water absorption									
30 minutes (A), %	44	40	49	47	47	37	48	51	39
24 hours (B), %	66	66	66	62	69	58	70	64	67
Ration A/B x 100	67	61	74	76	68	64	68	80	58

decreased or eliminated. Any differences should be shown in the yield figures—white weight to rough weight. On the whole, these figures are lower for the solvent-extract leathers than for the water-extract leathers, especially at medium or low temperatures. The most striking point in Table V is the high yield obtained with the 150° water extract, both on the rough and finished leather. The high yield obtained by the 10 per cent alcohol leather in the finished state is interesting, but as it was not confirmed by a corresponding high yield in the rough state, it must be considered as due to increase added in the oil wheel and of no particular significance in the actual tanning. In comparing Table V with Table II, it must be kept in mind that because of different methods of finishing, the weights of the dry rough leather are used in Table II, whereas in Table V the weights of the wet leather are used.

Table VI gives the chemical and physical data on the leathers in the second series of tests. As in the first series, most of the data require no comment, as they are normal for this type of leather. The most striking figure is the high degree of tannage, 87.7, for the 150° water-extract tannage, confirming the figures on yields in Table V.

Discussion

All leathers produced were satisfactory. They were firm, full, well-filled, and of good appearance. All canaigre extracts used by themselves gave good tannages, and a blend of canaigre extract with sulfited quebracho and chestnut extracts also gave good results.

One objection to canaigre is a tendency to mold. This may be prevented by the use of a small amount of preservative, which would have the disadvantage of preventing acid formation. However, acid formation in canaigre liquors is slight, and it would probably be more advantageous to add the required amount of acid rather than to depend upon fermentation. As canaigre extract would probably be used in blends with other materials, the bad effects of mold growth would be lessened, especially with the usual countercurrent method of tanning.

The penetration of tannin into the hide is more rapid with liquors made from water extracts than with liquors made from solvent extracts. In addition to delaying the tannage, slower penetration of the tannin has another effect. If the cut edges of the solvent-extract tanned leather are examined, dark layers or streaks are noticed, showing uneven tannage. Although this defect would almost certainly not occur in the countercurrent system of tannage used in commercial practice,, it indicates a difference in tanning properties between the two types of extracts.

The higher figures on yield and degree of tannage in Tables V and VI show clearly that the extract giving the best tanned leather was that prepared by extraction with water at 150° F. The extract prepared with acetone was inferior to those prepared with isopropyl alcohol. However, as the 18 per cent

alcohol extract was inferior to the 10 per cent alcohol extract, possibly the inferiority of the 25 per cent acetone extract was due more to the amount than to the nature of the solvent. The quality of the leather produced indicates that in solvent extraction the amount of solvent used should be kept to a minimum.

Sulfiting the extract improved penetration and color, but as expected, lowered the yield.

The question of sludge formation is important. The resulting loss of tannin necessitates a greater amount of tannin to produce the same amount of leather, as in the first round of the first series, or results in the production of a smaller amount of leather from the same amount of tannin, as shown in the yields of white weight to wet rough leather in the second series of tests. The results in the second round of the first series indicate that these liquors became stabilized by precipitation of sediment in the first round, and after this there was no further excessive deposition. In leathers tanned with the sludge-producing extracts, analysis showed that the tannin was less thoroughly combined than with other tannages. Furthermore, the sludge would require an extra process of scouring the leather. At present any comparison of cost of extracts produced by solvent extraction and those produced by water extraction only would be premature. However, it is certain that for solvent extraction to compare favorably with water extraction, greater recovery of tannin is necessary. Further work on the solvent extraction process is necessary to determine whether the extra tannin recovered is of advantage or whether it is lost during tannage.

Conclusions

Satisfactory leathers have been made from canaigre extracts prepared in various ways. Extracts prepared by water extraction gave better results than those prepared with a mixture of water and organic solvents. The best tanning results were obtained with an extract prepared with water at 150°F.

Acknowledgments

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Discussion

Wallace Windus: The significance of this research from the standpoint of national self-sufficiency in case of an emergency is apparent to all. The problem itself is particularly suitable for a government research laboratory, because it is of necessity a long-range project which requires patience and adequate financing.

Those of us who have visited any one of these Regional Research Laboratories know that the engineering facilities are impressive, indeed. And we are glad to be constantly reassured that the staff is definitely cost-conscious.

I have a few questions to lead off the discussion. I got the impression from Mr. Rogers' paper that the engineering staff has not yet decided on any one method of extraction. This paper is in the nature of a progress report, and I understand that further work will be done to decide on the most suitable extraction procedure from the standpoint of whether solvent or water and organic solvent combination. I would like to know if I am correct in that impression.

I also wonder about the amount of starch that still remains in the extract—whether there is a significant amount in the final extract and whether it has any disadvantages, if it is present.

It did appeal to me that the use of fermentation might not be necessary from a practical standpoint, since the purity is 58 per cent without fermentation, whereas it is only 69 per cent after fermentation, and some tanners might feel that the presence of fermentable sugars was desirable.

I was curous about the degree of tannage, but my question on that was answered. It is certainly adequate and satisfactory.

I would like to know something about the stability of the tannage in comparison with other tannages—whether the linkage of the canaigre tanning material with the protein is as strong, approximately, as with other vegetable materials.

Mr. Beebe: The extract was prepared in such a way as to eliminate the starch. Tests have shown that the amount of starch in the extract is insignificant.

As far as economy of the organic solvents as compared with water, that is still being worked on by the engineering Division and has not yet been settled. It is an engineering question. The cost of organic solvents is, of course, greater than water, but on the other hand, you get out more tannin. Whether one will balance the other or not is a question not yet decided.

We have not made any particular tests on the stability of the tannage yet.

Windus: I am thinking particularly of washing. Is it stable to washing and not readily washed out?

Beebe: It is stable. In working with canaigre extract, I would say that it is an average tannage. It does not have any particular advantage or disad-

vantage. It can be used as a blend with other materials. Particularly I would suggest that canaigre be used as most other tanning materials—blended with other tanning materials, such as quebracho and chestnut. You can use up to 40 per cent in blends.

We have not yet got to the point where we are producing the extract in high enough quantities to be used in a commercial tanning test, but we do expect to have a few hundred pounds in a little while, so that we can distribute a few pounds to anybody who is interested in working with that much.

MR. HILBERT: What is the percentage of starch in the original material that you have to remove?

ROGERS: In the dry, moisture-free roots the starch content will vary from around 25 per cent to 40 per cent.

DR. TU: I would like to know about the economic angle of a plantation of canaigre.

ROGERS: We have between twenty and thirty acres in canaigre, and expect to harvest about fourteen acres this summer. We anticipate that the yield will be in the neighborhood of 150 to 200 tons of fresh roots. We do not have specific data as to the cost of production. However, the canaigre can be planted with drills or planting machines in a manner similar to that used for other seeded crops, and the matured roots can be harvested with potato digging equipment. We see no reason why it should not be economically feasible. We hope to get cost data on some of that processing this year after the harvesting.

TU: How about the other parts of the plant? Do you find some other use for the other materials in the roots?

ROGERS: We are working on the utilization of both the starch and the sugar. At present, our fermentation process utilizes bacteria that remove sugars and raise the purity of the liquors, but the products of fermentation are not easily recovered. Studies of other fermentation procedures are underway which may produce a better by-product. The starch can be hydrolyzed and fermented. We hope to utilize both of these materials.

TU: Did you state that you get 37 per cent average tannin content from the roots?

ROGERS: Two different strains of roots have been observed so far. One type has yellow interiors and the tannin content varies from about 20 per cent to 25 per cent on a moisture-free basis. The other type of root has a reddish interior and its tannin content varies from 30 per cent up to as high as 54 per cent on a moisture-free basis. A further survey of wild plants is being made which may disclose other strains with different properties.

DR. BUECHLER: A number of years back, Mr. Roddy showed that tanning could be done with a solvent system. I wonder if Mr. Beebe has done work to see how well this material would tan in a solvent system?

BEEBE: We have not made any tests on solvent tanning.

BUECHLER: From the viewpoint of a national emergency, where it would be desirable to have a source of tannin in this country, it might be possible, instead of trying to get a water-soluble product, to sell your solvent extract of canaigre for solvent tannage. That would bring a lot of problems to the tanner, because he would have to have a solvent recovery system. However, he would save a lot of time in processing, because solvent tannage is so much faster. It would be a possibility, I think, to keep in mind.

BEEBE: I don't believe that solvent tannage has as yet proved commermercially successful. You can tan with solvents very rapidly, but whether you can get as high a yield of leather, I don't know. In time of a national emergency, there might be difficulty in obtaining solvents.

ROGERS: If any of you are interested in seeing heavy leather that has been tanned with canaigre extract we have some of it here.

WINDUS: In view of this discussion on the solvent tannage, if I may make one comment: I think it is very interesting as a theoretical laboratory procedure to study the mechanism of tanning, assuming that the reaction is analogous to what takes place in a water medium, of course. It is followed by water, so probably the mechanism of the tanning ends up the same, but I do not feel that it is a practical method of tanning. I don't think you will find tanners willing to consider all the expense and difficulties involved. A practical tanning agent to me must be water soluble.

DR. FLINN: Mr. Beebe gave tables showing three leathers tanned with extracts that were leached at three different temperatures—115, 150, and 180° F. Would the yield indicate that the one leached at 150° was significantly higher? Would you give an explanation for that?

BEEBE: I believe that the explanation is that at temperatures lower than 150°, there is insufficient extraction of tannin, while at higher temperatures there is interference by starch.

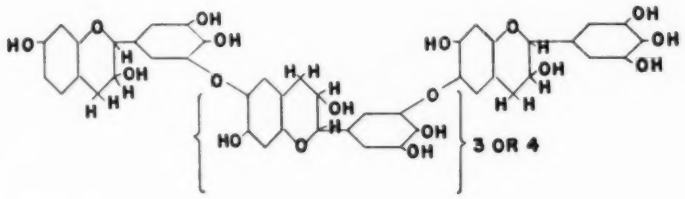
FLINN: Wouldn't that imply that since you had extracts leached at three different temperatures you were using extracts for tanning that had the chemical structure of the tannin molecule altered?

BEEBE: It may be possible. I don't have enough information to say that.

ABSTRACTS

A Contribution to the Structure of Black Wattle Tinnin. By D. G. Roux, *J. Soc. Lea. Trades Chemists*, **34**, 122(1950). Following the purification of black wattle tannin, studies on this tanning material were continued to include a detailed analysis of the acetylated and methylated derivatives of the purified material. A complete survey of all degradation products of black wattle tannin is first given, and their significance thoroughly discussed. From this consideration, the author concluded that the basic unit appears to be a 3:7:3′:4′:5′ - pentahydroxy - 2 - phenyl-chromane.

Considering all the evidence at hand the author concludes that black wattle tannin can be regarded as a mixture of polymers of the above unit formed by biochemical condensation for which he suggests a mechanism. A constant ratio in carbon, hydrogen and hydroxyl values has been found to exist throughout. Just over four hydroxyl groups per C_{15} unit have been shown present, while terminal methyl groups are absent. Slightly less than two ether oxygen groups per C_{15} unit appear to exist. The following represents a polymer of a single unit.



Chromatography of Proteins. By J. I. M. Jones and S. E. Michael. *Nature*, **165,** 685(1950). Through *J. Soc. Dyers and Colourists*, **66,** 394(1950). A simple method of location of proteins on a paper chromatogram or a cellulose column is described, based on the affinity of acid dyes for protein fibers and not for cellulose. The developed chromatogram is treated with a 0.05 per cent solution of a suitable dye, e. g. Solway Purple, containing 0.5 per cent sulphuric acid in a photographic developing dish at 50–90°C. The proteins show up as violet patches on the chromatogram. This dyeing property has been used for a study of the movement of proteins along the chromatogram using different liquids.

Hygroscopic and Wetting-Out Properties of Keratins. By O. Jacobi. *Kolloid Z.*, **114,** 88(1949), **116,** 48(1950). Through *J. Soc. Dyers and Colourists*, **66,** 338(1950). Experiments have shown that keratins, both untreated and degreased with fat solvents, are hydrophilic (i. e. they can be wetted out) and are also hygroscopic. Horn substances impregnated with keratin fats are, however, hydrophobic (i. e. do not wet out), but nevertheless are hygroscopic. Keratins extracted with water or alcohol, or water or alcohol followed by ether, are hydrophobic but hygroscopic. Horn substances, however, impregnated with water or alcohol extracts are hydrophilic.

Products of The Hydrolysis of Wool Keratin by Papain. By S. Blackburn, *Nature* **165,** 316(1950). Through *J. Soc. Dyers and Colourists*, **66,** 338(1950). Previous work by Middlebrook and Phillips [*J. S. D. C.*, **57,** 137(1941)] is now extended to the hydrolysis products themselves, when wool is treated with 2 per cent papain for 3½ hours at 65°C. in 1 per cent sodium bisulphite solution. The peptide precipitated by acidification to pH 4 has a disordered ξ-keratin type of structure and has a fairly long chain-length, while the peptides in soln. have a mean chain-length of 5 amino acid residues, and a two-dimensional chromatogram in collidine and phenol-ammonia showed glycine but no appreciable amount of other free amino acids. The following terminal amino acids were found among the peptides-leucine + isoleucine, valine, alanine, glycine, threonine, serine, and glutamic and aspartic acids. The enzyme thus liberates a large number of different amino groups from the peptide linkages, but with no high degree of specificity in its action on wool.

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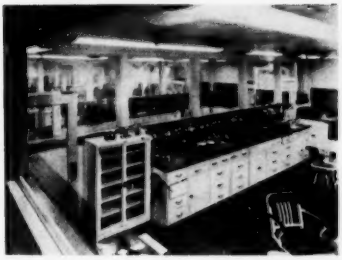
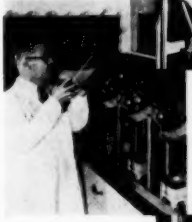


Tanners' samples, submitted for analysis are first carefully inspected.



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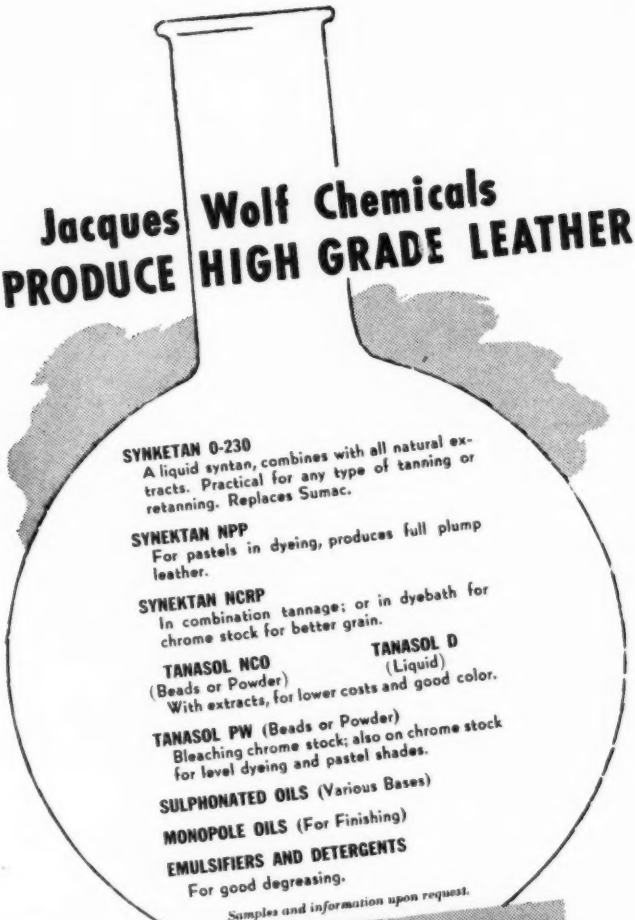

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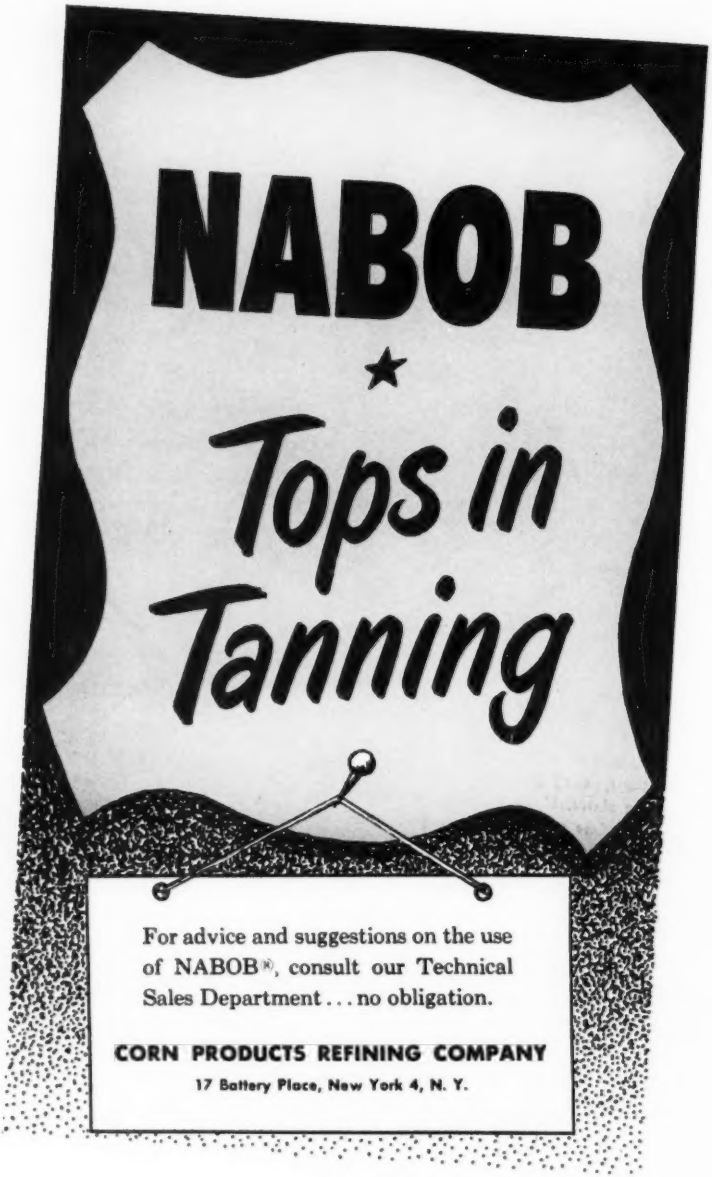
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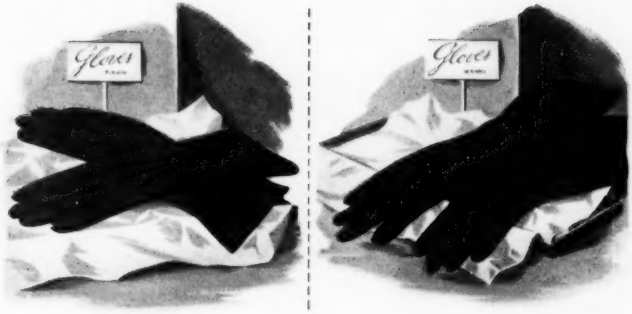

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